



WEC Energy Group, Inc.
231 W. Michigan St.
Milwaukee, WI 53203

February 23, 2018

Ms. Jenny Davison

Ms. Margaret Gielniewski

Ms. Sarah Rolfes

Mr. Pablo Valentin

Mr. Ross del Rosario

Ms. Leslie Patterson

Mr. Bill Ryan

United States Environmental Protection Agency

Region 5

77 West Jackson Boulevard

Chicago, Illinois 60604

**Subject: Administrative Settlement Agreement and Order on Consent
Multi-Site Quality Assurance Project Plan, Addendum No. 3, Revision 1 and
Response to Comments
The Peoples Gas Light and Coke Company, North Shore Gas, and
Wisconsin Public Service Corporation**

CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877 and V-W-08-C-917

Attached is our response to United Environmental Protection Agency (USEPA) comments on Addendum No. 3 of our Multi-Site Quality Assurance Project Plan (QAPP). We believe we have adequately addressed all of USEPA's comments and the QAPP addendum should be in an approvable format.

Hard copies and Sharefile downloads will be issued shortly per the distribution list on the enclosed cover letter.

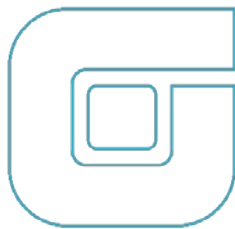
If you have any questions, please don't hesitate to contact me.

Sincerely,

A handwritten signature in blue ink, appearing to read 'Naren Prasad', written over a light blue horizontal line.

Naren M. Prasad, P.E., MPH

Principal Engineer – Environmental



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February 23, 2018

Mr. Naren Prasad
WEC Business Services, LLC
200 E. Randolph Drive, 21st Floor
Chicago, IL 60601

RE: Administrative Settlement Agreement and Order on Consent
Response to USEPA Comments on Multi-Site QAPP, Addendum No. 3
The Peoples Gas Light and Coke Company, North Shore Gas, and Wisconsin Public Service Corporation
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917
OBG Project Nos. 67848 and 67859

Dear Mr. Prasad:

O'Brien & Gere Engineers, Inc. (OBG) is providing the enclosed Revision 1 of Multi-Site Quality Assurance Project Plan (QAPP) Addendum No. 3 and responses to comments from United States Environmental Protection Agency (USEPA) dated January 4, 2018. These deliverables are being submitted for the Manufactured Gas Plant (MGP) Sites in the Multi-Site Program of The Peoples Gas Light and Coke Company (PGL), North Shore Gas (NSG), and Wisconsin Public Service Corporation (WPSC).

The QAPP Addendum No. 3, Revision 1 has incorporated USEPA comments. For ease of review, USEPA comments are presented below in *italics*, followed by the responses.

USEPA COMMENTS AND RESPONSES

USEPA Comment 1. The purpose of the QAPP Addendum No. 3, as stated in the first paragraph of the letter, is to incorporate changes or additions to the following elements of the program:

- *laboratories,*
- *vapor intrusion (VI) and soil gas evaluation,*
- *sample duplicate evaluation, and*
- *to address slow groundwater recharge in Chicago site monitoring wells.*

However, other than the update to the laboratories, this Addendum 3 does not appear to contain modifications to the field duplicate evaluation (previously modified in the Multi-Site QAPP Addendum No. 1 dated March 2012), or the slow groundwater recharge (previously presented in the Multi-Site QAPP Addendum No. 2 dated August 2014). Both of these addendums were attached, but have been previously approved by EPA. It is unclear whether there were new changes in Addendum 3 to the field duplicate or slow groundwater recharge sections, or if Addendums 1 and 2 were simply included for reference purposes. Please clarify.

RESPONSE: Multi-Site QAPP Addenda No. 1 and No. 2 were previously approved by USEPA. No modifications have been made to those addenda, they have been included for reference only. The cover letter language has been modified for clarity.



USEPA Comment 2. Organization Chart: Recommend inserting names in the darker blue boxes (WBS and Consultant) to be consistent with the approval page. Please also check pagination of the document, as QAPP Worksheet 6 starts on Page 10 rather than 9, and the organization chart is on page 11.

RESPONSE: The Organizational Chart has been modified to include the WEC Business Services LLC (WBS) and OBG staff who are identified on the workbook approval page; WBS Project Manager Naren Prasad, OBG Project Manager Sarah Meyer, and OBG Quality Assurance Officer Jennifer Hagen.

The pagination of the workbook has been corrected.

USEPA Comment 3. Table 1A: The text for some of the table footnotes appears to be cut-off and could not be reviewed.

RESPONSE: Table 1A footnotes have been modified for complete viewing.

USEPA Comment 4. Tables 1A-1D, 2A-2D, 3, 4, (Footnote "c"): Not all of the analytical methods referenced in the table refer to SW-846 Test Methods for Evaluating Solid Waste as indicated in table footnotes. Suggest adding the applicable method references.

RESPONSE: On each of the affected tables, Footnote "c" has been modified to refer to the laboratory standard operating procedures for specific details on analytical procedures. In addition, each method source, as abbreviated in the method name (e.g. SW846) has been defined either on the table or in the table notes (e.g. USEPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition).

USEPA Comment 5. Table series 1A-1D & 2A-2D have footnotes pertaining to "PQLs" (footnotes 1. and 2.). However, PQLs are not presented in the tables. Please explain the intent of presenting this PQL information in the table footnotes. Also, the PQL footnotes refer to "Surface water and sediment", while the content of the tables is either water or soil/sediment. For example, footnote No. 2 in tables 1A-1D (specific to the water matrix) includes 'normalizing to 1% TOC', 'sediments are reported on a dry weight basis', and 'RLs will vary based on the percent solids of the sediment samples'. The footnotes should be adjusted to pertain to the matrix being presented in the table.

RESPONSE: Reference to practical quantitation limits (PQL) on Tables 1A-D and 2A-D have been modified to reference screening levels (SL) and a discussion of PQLs is included in the cover letter. Neither PQLs or SLs are specified in the tables because the SLs used in the Multi-Site MGP Program are updated approximately every six months per the USEPA-approved Multi-Site Risk Assessment Framework. Footnote A was modified to state that prior to any project-specific field data collection, the selected lab's ability to achieve approved project-specific SLs, as provided in the current RAF Addendum, will be verified. In addition, footnotes were initially created to be consistent for each table series, however as requested, footnotes have been modified to be specific to each page within the series.

USEPA Comment 6. Multiple Tables (1A-1D and 2A-2D): "Pesticides" and "PCBs" are presented only by analyte class with a 'range' of laboratory limits. Suggest including the individual analytes for pesticides and PCBs consistent with the other analyte groups.

RESPONSE: As requested, individual analytes for pesticides and polychlorinated biphenyls (PCB) have been added to Tables 1A-D and 2A-D.

USEPA Comment 7. Multiple Tables (1A-1D): Hexavalent chromium is erroneously presented as "Chromium IV" and should be "Chromium VI". Table 1B appears to have been revised, but the edit is visible. (Another edit was observed in Table 2B, see the CAS Number for Chlordane.)

RESPONSE: As requested, references to hexavalent chromium and visible edits have been corrected.

USEPA Comment 8. Multiple Tables: The "Bold Underline Text" footnote indicates the laboratory will provide an alternate method than what is identified in the method column. In some tables "Alternate Methods Provided" are presented at the end of the table. The alternate methods provided at the end of each table do not appear to include some of the analytes indicated with the bold underline text. Please provide proposed laboratory methods.

RESPONSE: As requested, all alternate methods proposed have been added to the end of the tables. Also, a statement has been added to the footnotes of Tables 1A-1D and 2A-2D to indicate that additional analytical methods may be evaluated and proposed in Site-Specific Work Plans.

USEPA Comment 9. Tables 1D and 2D: The last column pair is labeled "Pace Grand Rapids", what lab location are the other three column pairs in these tables?

RESPONSE: Pace provided separate method detection limit (MDL) and reporting limit (RL) data for their Grand Rapids laboratory. The "other three columns" of Pace data on Tables 1D and 2D represent the capabilities of the entire Pace network. For clarity, the column titles have been modified to read "Pace Analytical Lab Network" and "Pace Analytical Grand Rapids Lab Only." Prior to any project-specific field data collection, the selected lab's ability to achieve approved project-specific SLs, as provided in the current RAF Addendum, will be verified.

USEPA Comment 10. Multiple Tables (especially tables 1D and 2D): Please provide further explanation when "na" (not applicable) is presented in the table in the RL or MDL fields, or update the fields containing "na" as appropriate.

RESPONSE: The "na" and "---" effectively have the same meaning on the tables of the original document: no data was provided. Either the lab chose to provide no data for that cell or no data was requested for that cell. For clarity, all cells for which no data are available have been filled with "---".

USEPA Comment 11. Tables 2A-2B: The first column of each table "Project Compound List" has an asterisk, defined in the footnotes as, "MDLs and RLs for alkylated PAHs are based on associated parent PAHs." Please place the "" in the MDL or RL column or on the actual compounds that are affected for clarity. Also, please justify the appropriateness of using lab limits for parent samples, but not specifying them for the specific alkylated PAHs.*

RESPONSE: Labs that provide analysis of both parent and alkylated polycyclic aromatic hydrocarbons (PAH) have provided MDLs and RLs for both parent and alkylated PAHs and these data are shown in Tables 1 and 2. RLs for parent PAHs are used for alkylated PAHs because the response factors of the parent PAHs are used to calculate the concentrations of alkylated PAHs. Likewise, the MDLs for the parent PAHs are used for the alkylated PAHs. Additional information on the basis for MDL and RL determinations for all analytes are provided in the lab standard operating procedures (SOP). As such, the "*" and related footnote have been removed from Tables 2A-2D and an additional footnote directing the reader to the laboratory SOP for additional information on laboratory methods and procedures has been added.

USEPA Comment 12. Table 2B: Why are the MDL and RL cells shaded grey for available cyanide?

RESPONSE: Shading has been removed.

USEPA Comment 13. Tables 3 and 7: The intent of tables 3 and 7 are unclear as they both appear to present the same information. The table 7 title indicates the table should present "Analytical Methods, Method Detection Limits and Reporting Limits" (similar to table series 1 and 2), however these laboratory limit values are not presented in table 7; while the multi-site QAPP Addendum No. 3 letter states tables 5 through 7 present the laboratories and range of services. Footnote A in Table 4 states the list of analytes, laboratory method and the method detection limit for each parameter are included in Tables 1-3 of the QAPP Addendum 3. Please clarify.

RESPONSE: Tables 5-7 are intended as a tool for project staff to provide a reference list and range of services for labs evaluated for analysis of water, soil and sediment, and air/soil gas/soil vapor, respectively. Table titles have been modified for clarity.

Table 3 has been modified to include the compound list for each analytical group and the current best achievable MDLs and RLs for labs enlisted to perform the listed analyses for the air/soil gas/soil vapor matrix. Prior to any project-specific field data collection, the selected lab's ability to achieve approved project-specific SLs, as provided in the current RAF Addendum, will be verified.

USEPA Comment 14. Tables 3 and 7: The compound list for each analytical group, MDLs, and RLs should be presented in either table 3 or 7.

RESPONSE: See response to comment 13. Table 3 has been modified to include the compound list for each analytical group and the current best achievable MDLs and RLs for labs enlisted to perform the listed analyses for the air/soil gas/soil vapor matrix.

USEPA Comment 15. Tables 3 and 7: There appears to be some discrepancies between tables 3 and 7. The table 7 title includes air, vapor, and soil gas matrices, while table 3 only indicates soil gas and vapor; and different method references including which methods include SIM.

RESPONSE: Tables 3 and 7 have been modified for consistency in matrix and methods.

USEPA Comment 16. Table 3, The text for some of the table footnotes appears to be cut-off and could not be reviewed. Where is the reference in the table located for footnote A?

RESPONSE: Table 3 footnotes have been modified for complete viewing and footnote A has been applied to the table heading.

USEPA Comment 17. Table 4 (first page): The row directly under Gasoline Range Organics does not have an analysis name presented in the first column. In the same row, please spell out or define the acronym "AK" in the "Minimum Volume/Size" column; AK is also presented in the "Method(s)" column for multiple rows in table 4.

RESPONSE: The format of Table 4 has been modified. Each laboratory has provided their preferred sample quantities, containers, preservation, minimum volume, and hold time to achieve method compliance and objectives for the analyses they are enlisted to perform. The term "AK" was not used on the revised tables.

USEPA Comment 18. Table 4: Many of the analytical method references in table 4 do not match the methods referenced in tables 1&2. Please revise and clarify.

RESPONSE: Table 4 was intended to include representative details for common sampling and analyses used for the program. For clarity, the format of Table 4 has been modified to match Tables 1 and 2. Each laboratory has provided their preferred sample quantities, containers, preservation, minimum volume, and hold time to achieve method compliance and objectives for the analyses for which they provided data on Tables 1 and 2. Sampling and analysis plan details will be confirmed with the selected lab on a site-specific basis prior to field data collection.

USEPA Comment 19. Table 4: the "Note for Soils", in the row for Total Petroleum Hydrocarbons is cut-off.

RESPONSE: See response to comment 17. The format of Table 4 has been modified. Each laboratory has provided their preferred sample quantities, containers, preservation, minimum volume, and hold time to achieve method compliance and objectives for the analyses they are enlisted to perform. The "Note for Soils" was not used on the revised tables.

USEPA Comment 20. Tables 5, 6, and 7: The table titles state, "Analytical Methods, Method Detection Limits (MDL), and Reporting Limits (RL)", however MDLs and RLs are not presented in these tables. Please revise accordingly.

RESPONSE: See response to Comment 13. Tables 5-7 are intended as a tool for project staff to provide a reference list and range of services for labs evaluated for analysis of water, soil and sediment, and air/soil gas/soil vapor, respectively. Table titles have been modified for clarity. Table 3 now includes the MDLs and RLs for the air/soil gas/soil vapor matrix.

USEPA Comment 21. Tables 5 and 6: It appears no labs are checked for Parameter/Method "Method 8021B (Act 201 List)" in table 5, or in the last two rows of table 6, "solid phase microextraction for PCBs" and "solid phase microextraction for other". Please revise or delete row.

RESPONSE: The requested modification has been made, rows with no data have been deleted from Tables 3, 5, 6, and 7.

USEPA Comment 22. Table 6: What is the intent of the expanded PAH list in footnote 2? There are 51 analytes listed in the footnote, 16 of which are not presented in the MDL and RL tables (1A-2D). Please revise and clarify.

RESPONSE: The expanded list of PAHs in the footnote included information used to screen labs for additional forensic analysis capabilities. If a longer, expanded list of PAHs is required to meet a future site-specific objective, these labs will be contacted for evaluation of those services. The footnote has been revised and simplified to indicate this.

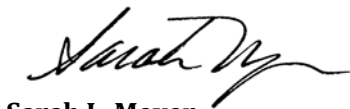
The information in footnote 1 on Table 6 was also used for lab screening, and the footnote has been revised and simplified.

USEPA Comment 23. Table 8, last row: There appears to be a typo, the reporting unit for Strength is presented as "pounds per foot inch" in the table. Please verify the units and abbreviation are correct.

RESPONSE: The table has been modified to indicate the unit for strength is tons per square foot (tsf).

Please contact the undersigned if you should have any questions regarding the content of this letter.

Very truly yours,
O'BRIEN & GERE ENGINEERS, INC.

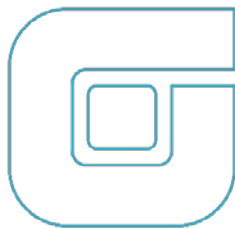


Sarah L. Meyer
Managing Scientist



Jennifer M. Hagen, PE
Senior Managing Engineer

Enc: Multi-Site QAPP Addendum No. 3, Revision 1



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February 23, 2018

Mr. Naren Prasad, PE, MPH
WEC Business Services, LLC
200 E. Randolph Drive, 21st Floor
Chicago, IL 60601

RE: Administrative Settlement Agreement and Order on Consent
Multi-Site QAPP, Addendum No. 3, Revision 1
The Peoples Gas Light and Coke Company, North Shore Gas, and Wisconsin Public Service Corporation
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917
OBG Project Nos. 67848 and 67859

Dear Mr. Prasad:

O'Brien & Gere Engineers, Inc. (OBG), is providing the following as Addendum No. 3, Revision 1 to the Multi-Site Quality Assurance Project Plan (QAPP) for the Manufactured Gas Plant (MGP) Sites in the Multi-Site Program of The Peoples Gas Light and Coke Company (PGL), North Shore Gas (NSG), and Wisconsin Public Service Corporation (WPSC). This addendum to the Multi-Site QAPP is necessary due to changes or additions in the laboratories and services used to execute field sample analysis and data assessment for the program.

The original QAPP (2007) and this document have been prepared as required in the Statement of Work (SOW) included with the Settlement Agreement and Administrative Order on Consent between USEPA and WPSC effective May 5, 2006; PGL effective June 5, 2007; NSG effective July 23, 2007; and PGL effective October 31, 2008.

For QAPP Addendum No. 3, Revision 1, capabilities and services offered by the following laboratories, that were included in the original QAPP, have been updated:

- Pace Analytical Services, Green Bay and Madison, WI; Minneapolis, MN; Pittsburgh, PA; and Grand Rapids, MI (formerly TriMatrix Laboratories, Inc.)
- Test America, University Park (Chicago), IL; Pittsburg, PA; Canton, OH; Knoxville and Nashville, TN; Burlington, VT; Sacramento, CA; Savannah, GA; and St. Louis, MO
- STAT Analysis Corporation, Chicago, IL
- Alpha Analytical, Mansfield, NH and Westborough, MA

For QAPP Addendum No. 3, Revision 1, the following laboratories are included as additional service providers for chemical analyses of environmental media and data evaluation:

- Brighton Analytical, Brighton, MI
- ESS, Cranston, RI
- Energy and Environmental Research Center (EERC), Grand Forks, ND
- Battelle, Norwell, MA
- Eurofins, Folsom, CA and Lancaster, PA



The original QAPP text remains the same. Therefore, this addendum is organized as a series of attachments and enclosures that include pertinent information as recommended in the Workbook for Uniform Federal Policy (UFP) for QAPPs and previously issued site-specific QAPP modifications.

The attached QAPP Addendum No. 3, Revision 1, Workbook includes a summary of the various modifications and improvements to the Multi-Site QAPP through a series of UFP QAPP worksheets and tables. The QAPP Addendum No. 3, Revision 1, Workbook includes:

- UFP QAPP Worksheets 1 *Title and Approval Page*, 2 *QAPP Identifying Information*, and 6 *Communication Pathways*. These worksheets provide the reviewer and approval signature page, guidance as to where the QAPP is being modified, and description of project communication, including a figure of the organizational chart.
- Tables 1, 2 and 3 *Multi-Site Program – Lowest Achievable Limits by Matrix* (similar to UFP QAPP Worksheet 15). These tables list the laboratories being added to the QAPP or updated, and their achievable method detection limits (MDL) and reporting limits (RL) for the water matrix, soil/sediment matrix, and air/soil gas/soil vapor matrix, respectively. The list of analytes included on these tables is a general list of compounds identified in the Multi-Site Risk Assessment Framework (RAF) and other documents for the MGP Multi-Site Program, plus some additional common compounds/analytes. Other analytes of interest can be listed in Site-Specific Work Plans and MDLs and RLs verified at that time. The laboratory limits may vary over time per lab performance tests, calibrations, and equipment updates. Note that practical quantitation limits (PQL) or the project-specific screening levels (SL) are not listed, because the SLs used in the Multi-Site MGP Program are updated approximately every six months per the USEPA-approved Multi-Site RAF. A RAF addendum will be submitted as these SLs change. As Multi-Site SLs are updated, each lab's ability to achieve new SLs is verified before new field data collection proceeds.
- Table 4 *Sampling and Analysis Summary* (similar to UFP QAPP Worksheet 19). On these tables, each laboratory has provided their preferred sample quantities, containers, preservation, minimum volume, and hold time to achieve method compliance and objectives for the analyses for which they provided data on Tables 1, 2 and 3.
- Tables 5-7 *Multi-Site Program Laboratory Services by Matrix*. These tables present a summary of laboratories and range of services it is anticipated they will provide for the Multi-Site Program. Laboratory standard operating procedures (SOP) for the laboratories and analyses/procedures listed on these tables are included in Enclosure A.
- Table 8 *Data Measurement Units for Field and Laboratory Measurements* has been updated to present agreed-upon sampling unit presentations for surface water and ground water, soil and sediment, air/soil gas/soil vapor.

Enclosures A-F to the Workbook are provided in electronic format, only. Each enclosure is listed and described below:

- Enclosure A - Laboratory/Validators Documentation. This enclosure consists of 10 separate sections:
 - » A1 to A9 are laboratory-specific information, including SOPs (new and updated), Quality Assurance Manuals (new or updated), and National Environmental Laboratory Accreditation (NELAC) and state certifications to be used to perform the work outlined in the Workbook. Some indicator parameters, geotechnical methods, sample preparation methods, disposal parameters and other SOPs are included in this enclosure as a reference only and may not be included on Tables 1-8. Note that laboratories consider some of their procedures and SOPs to be proprietary and prefer not to have them widely distributed and shared. If additional information is needed about any lab procedure, lab representatives can be made



available for discussion. In addition, it is anticipated that laboratories will update their SOPs as necessary and use the most current SOPs for work governed by the Multi-Site QAPP.

- » Enclosure A10 is new or updated qualification information for two data validators (MECx and Shepard Technical Services). The National Functional Guidelines for Data Review (NFG) are used as a basis for data validation. Data validators use the most current version of the NFG available.

In addition, the following previously-approved QAPP addenda are included by enclosure for reference only.

- Enclosure B - Multi-Site QAPP Addendum No. 1. For your reference, Multi-Site QAPP Addendum No. 1 is enclosed, it includes UFP QAPP Worksheet #28 for evaluation of field duplicate results for aqueous and non-aqueous media (Natural Resource Technology, Inc. (NRT), March 2012). This was originally submitted in response to USEPA comments on the Former Division Street Station Site-Specific Work Plan, Addendum No. 1. It was approved by USEPA on May 12, 2012.
- Enclosure C - Multi-Site QAPP Addendum No. 2. For your reference, Multi-Site QAPP Addendum No. 2 is enclosed, it is an updated laboratory usage and qualification addendum for Test America Laboratories (NRT, May 2012). This was originally submitted in preparation for removal action work at the Former Crawford Station MGP Site. It was approved by USEPA on March 12, 2012.

The remaining Enclosures D-G are USEPA-approved modifications relevant to the Multi-Site QAPP and quality assurance program. They are included for reference only as the modifications are being applied at the multi-site level:

- Enclosure D - Addendum No. 1, Revision 1, to Site-Specific Work Plan, Revision 1 for the Hough Place Station Former MGP Site (NRT, August 29, 2014). For your reference, this document includes updated laboratory qualifications for Test America and Pace Laboratories regarding use of USEPA Methods SW846-3510 and SW846-8270 for low-volume water samples (high volume injection) for analysis of polycyclic aromatic hydrocarbons (PAH), along with a modification to the groundwater sampling protocol for slow-recharging wells. It was approved by USEPA on September 4, 2014.
- Enclosure E - Appendix C of the Manitowoc Step II Site-Specific Work Plan, Revision 2 (NRT, April 30, 2012). For your reference, this document includes WBS's proposal and USEPA approval to use Meta Environmental, Inc. (Meta) to perform analysis of PAHs and alkylated PAHs in sediment and porewater by solid phase micro-extraction (SPME), gas chromatography (GC), mass spectrometry (MS) and selected ion monitoring (SIM). Meta's laboratory was purchased by Accutest Laboratories in 2013, but is no longer operating. The consulting portion of Meta now has a teaming alliance with ESS for laboratory analysis.
- Enclosure F - Email from Bob Paulson (WBS) to Margaret Gielniewski (USEPA) (August 3, 2017). For your reference, this email and its attachments provide qualifications, availability, and notification of intent to use EERC for SPME and bulk 34-PAH analysis. USEPA did not disagree with the choice to use EERC for this work.
- Enclosure G - Appendix A to the Milwaukee Solvay Coke and Gas Plant Site Engineering Evaluation and Cost Analysis Support Sampling Plan, Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan (NRT, October 2017). For your reference, this document includes WBS's proposal and USEPA approval on a Multi-Site QAPP Addendum to document site-specific QAPP elements for use at the Milwaukee Solvay Coke and Gas Plant Site. Addendum includes Pace Analytical Services laboratory PQLs and RLs for proposed soil and water analyses.

Laboratory-specific procedures and performance data meet the analytical requirements for the Multi-Site Program based on the risk-based SLs previously outlined in the approved RAF (Exponent, 2007) and subsequent addenda.



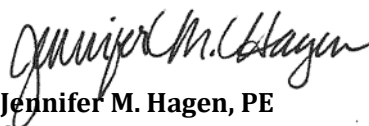
USEPA approval of QAPP Addendum No. 3, Revision 1 is requested. Future modifications to QAPP elements may be included in site-specific documents.

Please contact the undersigned if you should have any questions regarding the content of this letter.

Very truly yours,
O'BRIEN & GERE ENGINEERS, INC.



Sarah L. Meyer
Managing Scientist



Jennifer M. Hagen, PE
Senior Managing Engineer

Attachment:

QAPP Addendum No. 3, Revision 1 Workbook
Workbook Tables 1-8
Workbook Enclosures A1-A10 (electronic files, only)

For distribution to: Ross del Rosario, USEPA
Margaret Gielniewski, USEPA
Leslie Patterson, USEPA
Pablo Valentin, USEPA
William Ryan, USEPA
Sarah Rolfes, USEPA
Jenny Davison, USEPA
Paul Lake, IEPA
Kevin McKnight, WDNR
Tauren R. Beggs, WDNR
John Feeney, WDNR
Bill Fitzpatrick, WDNR
Liz Victor, WDNR
WDNR Regional Mailbox, DNRRRSER@wisconsin.gov (notification of FTP site upload)
WDNR FTP Site, <https://ftp.wi.gov/submittals/> (upload zipped files)
David Klatt, CH2M
Jennifer Knoepfle, CH2M
Bob Paulson, WBS
Frank Dombrowski, WBS
Mike Kierski, Exponent



Attachment - Workbook

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Tables 2A-2D	Multi-Site Program Soil/Sediment Matrix – Lowest Achievable Limits
Table 3	Multi-Site Program Air/Soil Gas/Soil Vapor Matrix – Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)
Table 4A	Sampling and Analysis Summary – Water Matrix
Table 4B	Sampling and Analysis Summary – Soil/Sediment Matrix
Table 4C	Sampling and Analysis Summary – Air/Soil Gas/Soil Vapor Matrix
Table 5	Multi-Site Program Laboratory Services – Water Analyses
Table 6	Multi-Site Program Laboratory Services – Soil/Sediment Analyses
Table 7	Multi-Site Program Laboratory Services – Air/Soil Gas/Soil Vapor Analyses
Table 8	Data Measurement Units for Field and Laboratory Measurements

Enclosures

Enclosure A	Laboratory/Validator Documentation (electronic files, only)
A1	Pace Analytical Laboratory
A2	Test America
A3	STAT Analysis
A4	Brighton Analytical Laboratories
A5	Alpha Analytical
A6	ESS
A7	Energy and Environmental Research Center
A8	Battelle
A9	Eurofins
A10	Validator SOQs
Enclosure B	– Multi-Site QAPP Addendum No. 1 (electronic files, only)
Enclosure C	– Multi-Site QAPP Addendum No. 2 (electronic files, only)
Enclosure D	– Addendum No. 1, Revision 1, to Site-Specific Work Plan, Revision 1 for the Hough Place Station Former MGP Site (Natural Resource Technology, Inc., August 29, 2014) (electronic files, only)
Enclosure E	- Appendix C of the Manitowoc Step II Site-Specific Work Plan, Revision 2 (Natural Resource Technology, Inc., April 30, 2012) (electronic files, only)
Enclosure F	- Email from Bob Paulson (WBS) to Margaret Gielniewski (USEPA) (August 3, 2017) (electronic files, only)

Title: Multi-Site QAPP, Addendum #3
Workbook
Revision Number: 1
Revision Date: 2/23/18
Page 2 of 12

Enclosure G – Appendix A to the Milwaukee Solvay Coke and Gas Plant Site Engineering
Evaluation and Cost Analysis Support Sampling Plan, Addendum to USEPA-Approved
Multi-Site Quality Assurance Project Plan (NRT, October 2017) (electronic files, only)

QAPP Worksheet #1

**(UFP-QAPP Manual Section 2.1)
Title and Approval Page**

Site Name: Various - Former Manufactured Gas Plant Sites for Wisconsin Public Service Corporation, The Peoples Gas Light and Coke Company, and North Shore Gas Under Administrative Settlement Agreement and Order on Consent (AOC) Nos. Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) V-W-'06-C-847, V-W-'07-C-869, V-W-'07-C-877, and V-W-'08-C-917

Document Title: Multi-Site Quality Assurance Project Plan (QAPP), Addendum No.3

Lead Organization: WEC Business Services, LLC (WBS)

Preparer's Name and Organizational Affiliation: Sarah L. Meyer, O'Brien & Gere Engineers, Inc. (OBG)

Preparer's Address, Telephone Number, and E-mail Address: 300 S. Wacker Drive, Suite 1300
Chicago, Illinois 60606
773.796.4606
Sarah.Meyer@obg.com

Preparation Date (Day/Month/Year): 3/November/2017

Lead Organization's Project Manager: _____
Signature

Printed Name/Organization/Date: Naren Prasad, WBS

Investigative Organization's Project Manager: _____
Signature 2/9/18

Printed Name/Organization/Date: Sarah Meyer, OBG

Investigative Organization's Project Quality Assurance (QA) Officer: _____
Signature 2/9/18

Printed Name/Organization/Date: Jennifer Hagen, OBG

Approval Signature: _____
Signature

Printed Name/Organization/Date: Ross del Rosario, USEPA Region 5 Project Coordinator

Approval Signature: _____
Signature

Printed Name/Organization/Date: Margaret Gielniewski, USEPA Region 5 Project Coordinator

Approval Signature: _____
Signature

Printed Name/Organization/Date: Leslie Patterson, USEPA Region 5 Project Coordinator

Approval Signature: _____
Signature

Printed Name/Organization/Date: Sarah Rolfes, USEPA Region 5 Project Coordinator

Approval Signature: _____
Signature

Printed Name/Organization/Date: Alida Roberman, USEPA Region 5 Superfund Division QA Team Leader

**QAPP Worksheet #2
(UFP-QAPP Manual Section 2.2.4)
QAPP Identifying Information**

Site Name/Project: Various - Former Manufactured Gas Plant Sites for Wisconsin Public Service Corporation, The Peoples Gas Light and Coke Company, and North Shore Gas

Site Location: Wisconsin and Illinois

Site Number/Code: Various - Under AOC Nos. CERCLA V-W-'06-C-847, V-W-'07-C-869, V-W-'07-C-877, and V-W-'08-C-917

1. Identify guidance used to prepare QAPP: USEPA Guidance for QAPPs and Uniform Federal Program (UFP) Worksheets
2. Identify regulatory program: CERCLA Superfund Alternative Sites Program
3. Identify approval entity: Not Applicable
4. Indicate whether the QAPP is a generic or a project-specific QAPP. Generic for use on sites under the Manufactured Gas Plant (MGP) Multi-Site Program
5. List dates of scoping sessions that were held: 6/6/17, 8/9/17, 8/18/17
6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title: Multi-Site QAPP - Quality Assurance Project Plan, Former Manufactured Gas Plant Sites, Volume 1 and 2, Revision 2 (September 2007)

Approval Date: 12/5/2007

7. List organizational partners (stakeholders) and connection with lead organization: Not Applicable
8. List data users: WBS and subconsultants, and USEPA Region 5
9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

Documents referenced in table below are:

AOC Nos. CERCLA V-W-'06-C-847, V-W-'07-C-869, V-W-'07-C-877, and V-W-'08-C-917

Multi-Site QAPP - Quality Assurance Project Plan, Former Manufactured Gas Plant Sites, Volume 1 and 2, Revision 2 (September 2007)

Multi-Site FSP – IBS 2008, Multi-Site Field Sampling and Analysis Plan, Former Manufactured Gas Plant Sites, Volume 1 and 2, Revision 4 (September 2008)

QAPP Worksheet #2
QAPP Identifying Information
Continued

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Worksheet #1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	Worksheet #2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Multi-Site QAPP Distribution List and Sign-off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	Multi-Site QAPP Section 1.2 and attached revised Figure 1
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	AOC V-W-'06-C-847, V-W-'07-C-869, V-W-'07-C-877, and V-W-08-C-917 or as specified in Site Specific Work Plan (project scoping) Multi-Site QAPP Section 1.3 (problem definition and background)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	Multi-Site QAPP Section 1.5 (quality objectives) Risk Assessment Framework Addendum, April 2011 (Exponent) and subsequent addendum or as specified in Site Specific Work Plans
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	As specified in Site Specific Work Plan
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Multi-Site QAPP Section 1.5 (quality objectives) or as specified in Site Specific Work Plan

Measurement/Data Acquisition		
<p>3.1 Sampling Tasks</p> <p>3.1.1 Sampling Process Design and Rationale</p> <p>3.1.2 Sampling Procedures and Requirements</p> <p>3.1.2.1 Sampling Collection Procedures</p> <p>3.1.2.2 Sample Containers, Volume, and Preservation</p> <p>3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures</p> <p>3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures</p> <p>3.1.2.5 Supply Inspection and Acceptance Procedures</p> <p>3.1.2.6 Field Documentation Procedures</p>	<ul style="list-style-type: none"> - Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/ SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table 	<p>Multi-Site QAPP Section 2. and Multi-Site FSP Section 4.1 (field sampling SOPs)</p> <p>Multi-Site QAPP Section 2.2.3 (field equipment calibration)</p> <p>Multi-Site QAPP Section 2.2.4 (supply inspection and acceptance)</p> <p>Multi-Site QAPP Section 2.9.1 (field documentation); Multi-Site FSP Appendix B (field forms)</p> <p>or as specified in Site Specific Work Plan</p>
<p>3.2 Analytical Tasks</p> <p>3.2.1 Analytical SOPs</p> <p>3.2.2 Analytical Instrument Calibration Procedures</p> <p>3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures</p> <p>3.2.4 Analytical Supply Inspection and Acceptance Procedures</p>	<ul style="list-style-type: none"> - Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table 	<p>As specified in Site Specific Work Plan; Attached Table and Multi-Site QAPP Section 2.0</p>
<p>3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures</p> <p>3.3.1 Sample Collection Documentation</p> <p>3.3.2 Sample Handling and Tracking System</p> <p>3.3.3 Sample Custody</p>	<ul style="list-style-type: none"> - Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal 	<p>Multi-Site QAPP Section 2.3 and Attachment 1</p> <p>Multi-Site FSP Section 5</p>

3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	Multi-Site QAPP Section 2.5 or as specified in Site Specific Work Plan
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	Multi-Site QAPP Section 2.3.2.3 Multi-Site FSP Section 4.9
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	Multi-Site QAPP Section 3.1
4.2 QA Management Reports	- QA Management Reports Table	Multi-Site QAPP Section 3.2.1
4.3 Final Project Report		Multi-Site QAPP Section 3.2.2 and 3.2.3
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	AOC V-W-11-C-9 Multi-Site QAPP Section 4 And as specific in Site Specific Work Plan

5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		As specified in Site Specific Work Plan
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QAPP Worksheet #6

(UFP-QAPP Manual Section 2.4.2)

Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modification to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Change to sampling plan (sample locations, numbers, or analytes) due to site-specific/field conditions See Figure 1	O'Brien & Gere Engineers, Inc.	Site Project Manager	See Site-Specific Work Plan	Notify USEPA, and request approval as appropriate, prior to authorizing sample collection or laboratory analysis

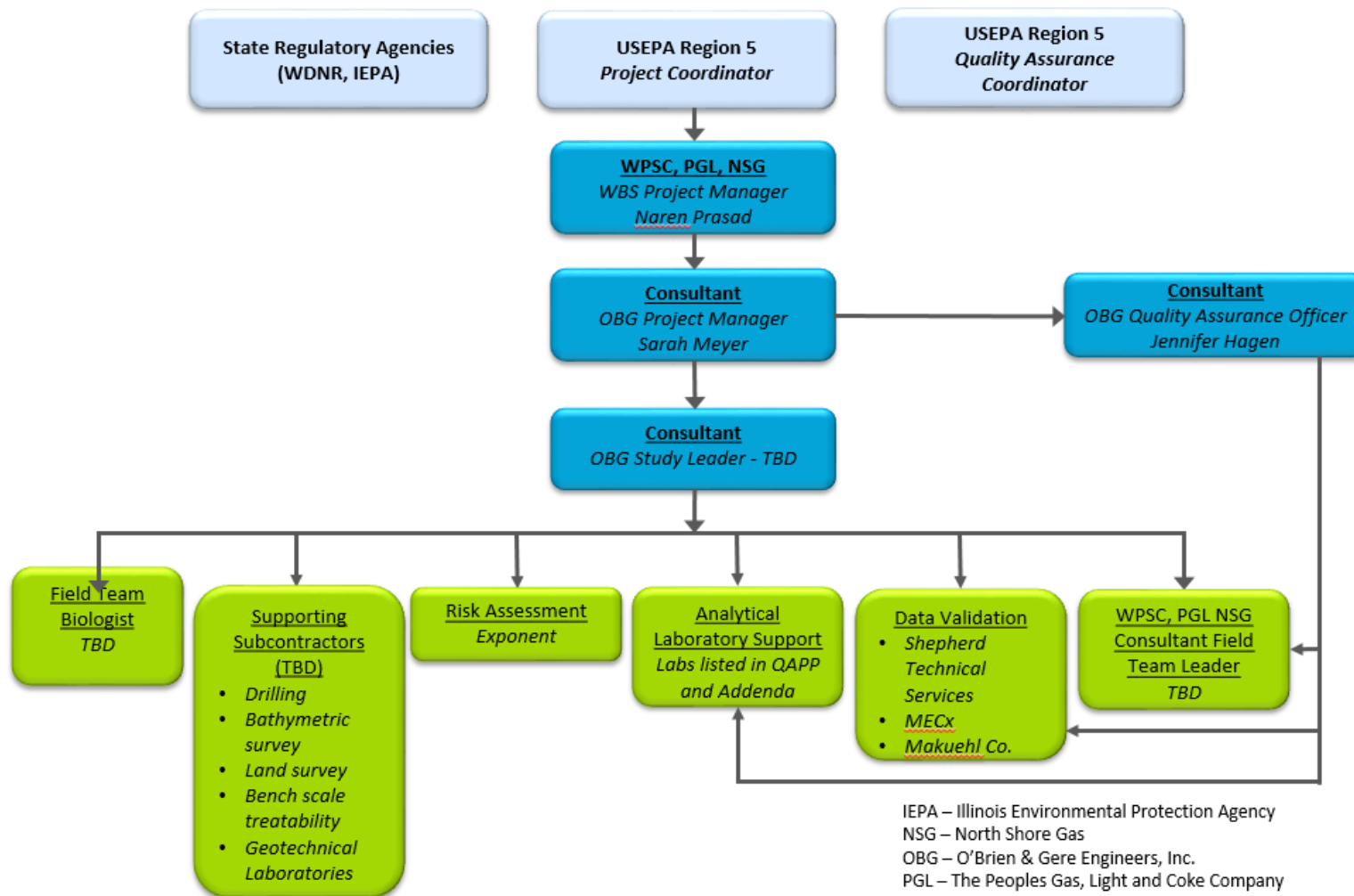


Figure 1 - Organizational Chart
Multi-Site QAPP, Addendum 3
WPSC, PGL and NSG

IEPA – Illinois Environmental Protection Agency
NSG – North Shore Gas
OBG – O’Brien & Gere Engineers, Inc.
PGL – The Peoples Gas, Light and Coke Company
TBD – To be determined, site-specific selections
USEPA – United States Environmental Protection Agency
WBS – WEC Business Services, LLC
WDNR – Wisconsin Department of Natural Resources
WPSC – Wisconsin Public Service Corporation



Tables



Table 1A. Multi-Site Program Water Matrix - Lowest Achievable Limits - Alpha, Brighton, STAT, Battelle and ESS
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Alpha - Lowest Achievable Limits		Brighton - Lowest Achievable Limits		STAT - Lowest Achievable Limits		Battelle - Lowest Achievable Limits		ESS - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Volatile Organic Compounds												
Benzene	71-43-2	SW846-8260B/8260C/ 8021B	0.159	0.500	0.35	1	0.2	5	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B/8260C/ 8021B	0.168	0.500	0.21	1	0.3	5	---	---	---	---
Toluene	108-88-3	SW846-8260B/8260C/ 8021B	0.161	0.750	0.39	1	0.4	5	---	---	---	---
Xylenes, total	1330-20-7	SW846-8260B/8260C/ 8021B	0.33	1.00	0.43	3	1	15	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B/8260C/ 8021B	0.174	2.50	0.19	1	0.2	5	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B/8260C/ 8021B	0.191	2.50	0.25	1	0.2	5	---	---	---	---
Methyl-Tert-Butyl Ether	1634-04-4	SW846-8260B/8260C/ 8021B	0.16	1.00	0.99	1	0.3	5	---	---	---	---
1,1,2,2-Tetrachloroethane	79-34-5	SW846-8260B/8260C/ 8021B	0.144	0.500	0.77	1	0.1	5	---	---	0.1	0.5
Tetrachloroethene	127-18-4	SW846-8260B/8260C/ 8021B	0.181	0.500	0.79	1	0.3	5	---	---	0.2	1
Trichloroethene	79-01-6	SW846-8260B/8260C/ 8021B	0.175	0.500	0.64	1	0.3	5	---	---	0.2	1
Vinyl Chloride	75-01-4	SW846-8260B/8260C/ 8021B	0.0699	1.00	0.28	1	0.2	2	---	---	0.2	1
Chloroform	67-66-3	SW846-8260B/8260C/ 8021B	0.162	0.750	1.33	10	0.1	5	---	---	0.2	1.0
Isopropylbenzene (aka Cumene)	98-82-8	SW846-8260B/8260C/ 8021B	0.187	0.500	1.03	10	0.4	5	---	---	0.1	1.0
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	35.3	500	10	100	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	3.048	50.0	10	100	---	---	---	---	---	---
Semivolatile Organic Compounds												
Naphthalene	91-20-3	SW846-8270C/D/SIM PAH	0.00197	0.010	0.10	5	0.1	1	0.0048	0.005	0.058	0.572
Nitrobenzene	98-95-3	SW846-8270C/D	0.102	0.500	0.65	5	0.03	1	---	---	3.2	10.0
C1-naphthalenes	---	SW846-8270C/D/SIM PAH	0.00197	0.010	---	---	---	---	0.0048	0.005	0.058	0.572
C2-naphthalenes	---	SW846-8270C/D/SIM PAH	0.00197	0.010	---	---	---	---	0.0048	0.005	0.058	0.572
C3-naphthalenes	---	SW846-8270C/D/SIM PAH	0.00197	0.010	---	---	---	---	0.0048	0.005	0.058	0.572
C4-naphthalenes	---	SW846-8270C/D/SIM PAH	0.00197	0.010	---	---	---	---	0.0048	0.005	0.058	0.572
Acenaphthylene	208-96-8	SW846-8270C/D/SIM PAH	0.002	0.010	0.18	5	0.03	1	0.00112	0.005	0.079	0.572
Acenaphthene	83-32-9	SW846-8270C/D/SIM PAH	0.00128	0.010	0.26	5	0.04	1	0.0013	0.005	0.030	0.572
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.00181	0.010	0.31	5	0.04	1	0.0011	0.005	0.105	0.572
Fluorene	86-73-7	SW846-8270C/D/SIM PAH	0.00177	0.010	0.30	5	0.05	1	0.00111	0.005	0.072	0.572
C1-fluorenes	---	SW846-8270C/D/SIM PAH	0.00177	0.010	---	---	---	---	0.00111	0.005	0.072	0.572
C2-fluorenes	---	SW846-8270C/D/SIM PAH	0.00177	0.010	---	---	---	---	0.00111	0.005	0.072	0.572
C3-fluorenes	---	SW846-8270C/D/SIM PAH	0.00177	0.010	---	---	---	---	0.00111	0.005	0.072	0.572
Phenanthrene	85-01-8	SW846-8270C/D/SIM PAH	0.0012	0.010	0.19	5	0.07	1	0.00227	0.005	0.060	0.572
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.00181	0.010	0.31	5	0.04	1	0.0011	0.005	0.105	0.572
C1-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.0012	0.010	---	---	---	---	0.00227	0.005	0.060	0.572
C2-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.0012	0.010	---	---	---	---	0.00227	0.005	0.060	0.572
C3-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.0012	0.010	---	---	---	---	0.00227	0.005	0.060	0.572
C4-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.0012	0.010	---	---	---	---	0.00227	0.005	0.060	0.572
Fluoranthene	206-44-0	SW846-8270C/D/SIM PAH	0.00178	0.010	0.19	5	0.02	1	0.00144	0.005	0.038	0.572
Pyrene	129-00-0	SW846-8270C/D/SIM PAH	0.00182	0.010	0.69	5	0.03	1	0.00131	0.005	0.060	0.572
C1-pyrene/fluoranthenes	---	SW846-8270C/D/SIM PAH	0.00182	0.010	---	---	---	---	0.00131	0.005	0.060	0.572
Benzo(a)anthracene	56-55-3	SW846-8270C/D/SIM PAH	0.00116	0.010	0.34	2	0.04	0.1	0.00126	0.005	0.108	0.572
Chrysene	218-01-9	SW846-8270C/D/SIM PAH	0.00126	0.010	0.23	5	0.06	0.1	0.0013	0.005	0.082	0.572
C1-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.00126	0.010	---	---	---	---	0.0013	0.005	0.082	0.572
C2-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.00126	0.010	---	---	---	---	0.0013	0.005	0.082	0.572
C3-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.00126	0.010	---	---	---	---	0.0013	0.005	0.082	0.572
C4-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.00126	0.010	---	---	---	---	0.0013	0.005	0.082	0.572
Benzo(b)fluoranthene	205-99-2	SW846-8270C/D/SIM PAH	0.00147	0.010	0.60	2	0.03	0.1	0.00127	0.005	0.131	0.572
Benzo(k)fluoranthene	207-08-9	SW846-8270C/D/SIM PAH	0.00149	0.010	0.50	5	0.06	0.1	0.00118	0.005	0.089	0.572
Benzo(a)pyrene	50-32-8	SW846-8270C/D/SIM PAH	0.00215	0.010	0.32	2	0.06	0.1	0.00142	0.005	0.183	0.572
Perylene	198-55-0	SW846-8270C/D/SIM PAH	0.00183	0.010	---	---	---	---	0.00147	0.005	0.058	0.572
Benzo(e)pyrene	192-97-2	SW846-8270C/D/SIM PAH	0.00131	0.010	---	---	---	---	0.00105	0.005	0.103	0.572
Indeno(1,2,3-cd)pyrene	193-39-5	SW846-8270C/D/SIM PAH	0.00246	0.010	0.48	5	0.06	0.1	0.00166	0.005	0.175	0.572
Dibenzo(a,h)anthracene	53-70-3	SW846-8270C/D/SIM PAH	0.00294	0.010	0.66	2	0.05	1	0.00132	0.005	0.249	0.572
Benzo(g,h,i)perylene	191-24-2	SW846-8270C/D/SIM PAH	0.00265	0.010	0.32	5	0.04	1	0.00113	0.005	0.176	0.572
1-Methylnaphthalene	90-12-0	SW846-8270C/D/SIM PAH	0.00195	0.010	---	---	0.04	1	0.00338	0.005	0.045	0.572
2-Methylnaphthalene	91-57-6	SW846-8270C/D/SIM PAH	0.0023	0.010	0.57	5	0.03	1	0.00269	0.005	0.040	0.572
1,2-Dichloroethene	540-59-0	SW846-8270C/D/SIM PAH	---	---	---	---	0.2	5	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Alpha - Lowest Achievable Limits		Brighton - Lowest Achievable Limits		STAT - Lowest Achievable Limits		Battelle - Lowest Achievable Limits		ESS - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
cis-1,2-Dichloroethene	156-59-2	SW846-8270C/D/SIM PAH	---	---	---	---	0.2	5	---	---	---	---
trans-1,2-Dichloroethene	156-60-5	SW846-8270C/D/SIM PAH	---	---	---	---	0.2	5	---	---	---	---
Bis(2-Ethylhexyl)Phthalate	117-81-7	SW846-8270C/D/SIM PAH	0.910	3.00	0.66	5	0.2	5	---	---	2.0	6.0
p-Isopropyltoluene	99-87-6	SW846-8270C/D/SIM PAH	---	---	---	---	0.4	5	---	---	0.286	0.572
Dichloromethane	75-09-2	SW846-8270C/D/SIM PAH	---	---	---	---	0.2	5	---	---	---	---
n-Nitrosodiphenylamine	86-30-6	SW846-8270C/D/SIM PAH	0.072	0.500	0.39	5	0.9	5	---	---	3.3	10.0
Carbazole	86-74-8	SW846-8270C/D/SIM PAH	0.00154	0.010	0.57	10	0.02	0.1	---	---	3.3	10.0
Dibenzofuran	132-64-9	SW846-8270C/D/SIM PAH	0.00182	0.010	0.53	5	0.3	1	0.00116	0.005	0.0323	0.572
Phenols												
2,4-Dichlorophenol	120-83-2	SW846-8270C/D	0.769	5	0.50	5	1.7	5	---	---	3.0	10.0
2,4-Dimethylphenol	105-67-9	SW846-8270C/D	1.64	5	0.36	5	1.3	5	---	---	6.8	20.0
2-Methylphenol (o-cresol)	95-48-7	SW846-8270C/D	1.02	5	0.57	5	1.3	5	---	---	3.2	10.0
3&4-Methylphenol (m, p-cresol)	106-44-5	SW846-8270C/D	1.11	5	0.57	5	2.8	5	---	---	6.2	20.0
4-Methylphenol	106-44-5	SW846-8270C/D	---	---	0.55	5	2.8	5	---	---	---	---
Phenol	108-95-2	SW846-8270C/D	1.89	5	0.08	5	0.8	5	---	---	3.3	10.0
Phenolics	Multiple	SW846 9066	---	---	5.6	10	9.6	25	---	---	---	---
Pesticides												
Aldrin	309-00-2	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
α-BHC	319-84-6	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
β-BHC	319-85-7	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
δ-BHC	319-86-8	SW846 8081B	0.0005	0.0005	---	---	---	---	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081B	0.0005	0.0005	---	---	---	---	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081B	0.0005	0.0005	---	---	---	---	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	0.025	0.025	0.01	0.02	---	---	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	0.327	2.5	---	---	---	---	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
4,4'-DDE	72-55-9	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Diallate	2303-16-4	SW846 8081B			0.01	0.02	---	---	---	---	---	---
Dieldrin	60-57-1	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Endosulfan I	959-98-8	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Endrin	72-20-8	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Endrin ketone	53494-70-5	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Heptachlor	76-44-8	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	0.0005	0.0005	0.01	0.02	---	---	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	0.002	0.002	0.01	0.02	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	7.84	20	0.01	0.02	---	---	---	---	---	---
Isodrin	465-73-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---
Methoxychlor	72-43-5	SW846 8081B	0.005	0.005	0.01	0.02	---	---	---	---	---	---
Toxaphene	8001-35-2	SW846 8081B	0.025	0.025	---	---	---	---	---	---	---	---
PCBs												
Aroclor 1016	12674-11-2	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
Aroclor 1260	11096-82-5	SW846 8082A	0.02	0.02	0.05	0.2	---	---	---	---	---	---
PCB Congeners												
2-Chlorobiphenyl	2051-60-7	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	0.0005	0.001	---	---	---	---	---	---	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---

Table 1A

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Alpha - Lowest Achievable Limits		Brighton - Lowest Achievable Limits		STAT - Lowest Achievable Limits		Battelle - Lowest Achievable Limits		ESS - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	0.0005	0.001	---	---	---	---	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	0.0005	0.001	---	---	---	---	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	0.0005	0.001	---	---	---	---	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	0.00025	0.0005	---	---	---	---	---	---	---	---
Inorganics												
Aluminum	7429-90-5	SW846-6020A/ 6010/ EPA method 200.7	3.27	10.0	0.65	50	4.15	20	---	---	---	---
Antimony	7440-36-0	SW846-6020A/ 6010/ EPA method 200.7	0.429	4.00	0.32	5	0.92	3	---	---	---	---
Arsenic, total	7440-38-2	SW846-6020A/ 6010/ EPA method 200.7	0.165	0.500	0.22	1	0.18	2	---	---	---	---
Barium, total	7440-39-3	SW846-6020A/ 6010/ EPA method 200.7	0.173	0.500	0.14	5	0.59	2	---	---	---	---
Cadmium	7440-43-9	SW846-6020A/ 6010/ EPA method 200.7	0.0599	0.200	0.19	0.2	0.25	1	---	---	---	---
Chromium III	---	SW846-6020A/ 6011	---	---	---	5	---	---	---	---	---	---
Chromium VI	18540-29-9	SW846-6020A/ 6012/7196A/3060A/SM3500Cr-B	---	10	0.5	5	2	10	---	---	---	---
Chromium, total	7440-47-3	SW846-6020A/ 6010/ EPA method 200.7	0.178	0.500	0.11	2	0.3	20	---	---	---	---
Copper, total	7440-50-8	SW846-6020A/ 6010/ EPA method 200.7	0.384	1.00	0.63	4	0.34	5	---	---	---	---
Cyanide, total	57-12-5	SW846-9012B	1.80	5.00	0.94	5	35	250	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	---	---	0.41	2	---	---	---	---	---	---
Cyanide, amenable	57-12-5	SW846 9012B/9014	0.200	0.200	0.94	5	2	5	---	---	---	---
Cyanide, dissociable	57-12-5	SM 4500CN	---	---	---	---	2	5	---	---	---	---
Iron, dissolved	7439-89-6	SW846-6020A/ 6010/ EPA method 200.7	19.1	50.0	5.8	20	7.76	50	---	---	---	---
Iron, total	7439-89-6	SW846-6020A/ 6010/ EPA method 200.7	19.1	50.0	7.7	20	7.76	50	---	---	---	---
Lead	7439-92-1	SW846-6020A/ 6010/ EPA method 200.7	0.343	1.0	0.11	1	0.23	1	---	---	---	---
Manganese, total	7439-96-5	SW846-6020A/ 6010/ EPA method 200.7	0.440	1.0	0.12	5	0.28	2	---	---	---	---
Mercury	7439-97-6	SW846-7470A/7471B/7474	0.010	0.050	0.021	0.2	0.6	20	---	---	---	---
Nickel, total	7440-2-0	SW846-6020A/ 6010/ EPA method 200.7	0.556	2.00	0.53	20	0.24	2	---	---	---	---
Selenium, total	7782-49-2	SW846-6020A/ 6010/ EPA method 200.7	1.73	5.00	0.40	2	0.3	2	---	---	---	---
Silver, total	7440-22-4	SW846-6020A/ 6010/ EPA method 200.7	0.163	0.400	0.03	0.2	0.14	2	---	---	---	---
Thallium, total	7440-28-0	SW846-6020A/ 6010/ EPA method 200.7	0.143	0.500	0.1	2	0.12	1	---	---	---	---
Vanadium	7440-62-2	SW846-6020A/ 6010/ EPA method 200.7	1.57	5.00	0.08	10	0.47	2	---	---	---	---
Zinc, total	7440-66-6	SW846-6020A/ 6010/ EPA method 200.7	3.41	10.0	1.07	4	1.11	10	---	---	---	---
Other												
Alkalinity as CaCO ₃	3812-32-6	SM 2320B	---	2000	1000	5000	2400	20000	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	23.0	75.0	8	10	500	1000	---	---	---	---
Biochemical Oxygen Demand (BOD)	---	SM 5210B	---	2000	---	2000	410	2000	---	---	---	---
Chloride	16887-00-6	SM 4500-Cl E	200	1000	0.11	1000	---	---	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	5099	20000	2000	3000	7300	20000	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	17.0	200	0.06	100	90	500	---	---	---	---
Hardness	---	SW846 6010/6020	<u>0.2</u>	<u>0.7</u>	500	.5000	200	1000	---	---	---	---
Methane	74-82.8	RSK-175	0.50	0.50	1	2	---	---	---	---	---	---
Nitrate	14797-55-8	EPA Method 353.2/SM4500NO ₃ -F/ SM4500NO ₂ -B	32.8	100	0.003	50	39	200	---	---	---	---
Nitrogen, Nitrate & Nitrite	14797-55-8 &14797-65-0	EPA Method 353.2/SM4500NO ₃ -F/ SM4500NO ₂ -B	32.8	100	0.003	50	39	200	---	---	---	---
Oil & Grease	---	EPA Method 1664A	4000	4000	---	5000	460	5000	---	---	---	---
pH	---	EPA Method 150.1/ SM 9040/ SM 9045	---	---	---	---	---	---	---	---	---	---
Phosphate	14265-44-2	EPA Method 365.1	<u>0.003</u>	<u>0.01</u>	10	60	---	---	---	---	---	---
Residue, Non-filterable Total Suspended Solids (TSS)	---	SM 2540D	---	---	5100	10000	3100	7500	---	---	---	---
Sulfate	14808-79-8	SM 4500 SO4 E	1370	10000	0.38	1000	700	5000	---	---	---	---
Sulfur	7704-34-9	SW846 6010	---	---	100	1000	---	---	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151/8321A/8270C/D	---	---	---	---	0.16	0.2	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060	114	500	0.39	1000	180	500	---	---	---	---
Alternate Methods Provided												
Hardness	---	SM 2340B	0.2	0.7	---	---	---	---	---	---	---	---
Phosphate	14265-44-2	SM 4500P-E	0.003	0.01	---	---	---	---	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8260C	0.327	2.5	---	---	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8270D	7.84	20	---	---	---	---	---	---	---	---

O: SLM C: SSW F: SLM

Notes:
aka = also known as
CAS = Chemical Abstracts Service
CaCO₃ = Calcium Carbonate
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136
MDL = Method Detection Limit
MGP = Manufactured Gas Plant
OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl
RAF = Multi-Site Risk Assessment Framework
RCRA = Resource Conservation and Recovery Act
RL = Reporting Limit
RSK-175 = RSKSOP-175, 2006, prepared for use of the Ground Water and Ecosystems Restoration Division of USEPA
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP = Standard operating procedure
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
TPH = Total Petroleum Hydrocarbons
USEPA or EPA = United States Environmental Protection Agency
VOA = volatile organic analyte/analysis
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
µg/L = micrograms per liter
--- = Lab does not offer analysis, analysis was not requested, no data
Bold Underline Text = Lab will provide an alternate method than what is identified in method column

^A Prior to any project-specific field data collection, the selected lab’s ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for surface water and groundwater. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 1B. Multi-Site Program Water Matrix - Lowest Achievable Limits - Test America Only
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits	
			Chicago		Pittsburgh		Nashville		Knoxville		Canton	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Volatile Organic Compounds												
Benzene	71-43-2	SW846-8260B	0.146	0.5	---	---	---	---	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B	0.183	0.5	---	---	---	---	---	---	---	---
Toluene	108-88-3	SW846-8260B	0.152	0.5	---	---	---	---	---	---	---	---
Xylenes, total	1330-20-7	SW846-8260B	0.219	1.0	---	---	---	---	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B	0.254	1.0	---	---	---	---	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B	0.358	1.0	---	---	---	---	---	---	---	---
Methyl-Tert-Butyl Ether	1634-04-4	SW846-8260B	0.394	1.0	---	---	---	---	---	---	---	---
1,1,2,2-Tetrachloroethane	79-34-5	SW846-8260B	0.398	1.0	---	---	---	---	---	---	---	---
Tetrachloroethene	127-18-4	SW846-8260B	0.37	1.0	---	---	---	---	---	---	---	---
Trichloroethene	79-01-6	SW846-8260B	0.164	0.5	---	---	---	---	---	---	---	---
Vinyl Chloride	75-01-4	SW846-8260B	0.204	0.5	---	---	---	---	---	---	---	---
Chloroform	67-66-3	SW846-8260B	0.4	2.0	---	---	---	---	---	---	---	---
Isopropylbenzene (aka Cumene)	98-82-8	SW846-8260B	0.4	1.0	---	---	---	---	---	---	---	---
1,2-Dichloroethene-VOA	540-59-0	SW846-8260B	0.409	2.0	---	---	---	---	---	---	---	---
cis-1,2-Dichloroethene-VOA	156-59-2	SW846-8260B	0.409	1.0	---	---	---	---	---	---	---	---
trans-1,2-Dichloroethene-VOA	156-60-5	SW846-8260B	0.349	1.0	---	---	---	---	---	---	---	---
p-Isopropyltoluene-VOA	99-87-6	SW846-8260B	0.362	1.0	---	---	---	---	---	---	---	---
Dichloromethane-VOA (Methylene Chloride)	75-09-2	SW846-8260B	1.63	5.0	---	---	---	---	---	---	---	---
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	10.0	30.0	---	---	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	32.5	100.0	---	---	---	---	---	---	---	---
Semivolatile Organic Compounds			Routine PAHs/SVOCs		LL PAHs/SVOCs		Alkylated/Forensic PAHs		LL Alkylated PAHs			
			SW846-8270D		SW846-8270D		SW846-8270D SIM		GC/MS Isotope Dilution			
Naphthalene	91-20-3	Refer to Column Header	0.247	0.800	0.175	0.190	0.0500	0.0500	0.016	0.05	---	---
Nitrobenzene	98-95-3	Refer to Column Header	0.359	0.8	0.134	2	---	---	---	---	---	---
C1-naphthalenes	---	Refer to Column Header	---	---	---	---	**	**	**	**	---	---
C2-naphthalenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C3-naphthalenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C4-naphthalenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
Acenaphthylene	208-96-8	Refer to Column Header	0.214	0.800	0.0602	0.190	0.05	0.05	0.00015	0.01	---	---
Acenaphthene	83-32-9	Refer to Column Header	0.247	0.800	0.0724	0.190	0.05	0.05	0.0024	0.01	---	---
Anthracene	120-12-7	Refer to Column Header	0.267	0.800	0.0558	0.190	0.05	0.05	0.00071	0.01	---	---
Fluorene	86-73-7	Refer to Column Header	0.195	0.800	0.112	0.190	0.05	0.05	0.0015	0.01	---	---
C1-fluorenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C2-fluorenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C3-fluorenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
Phenanthrene	85-01-8	Refer to Column Header	0.241	0.800	0.129	0.190	0.05	0.05	0.011	0.02	---	---
C1-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C2-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C3-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
C4-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
Fluoranthene	206-44-0	Refer to Column Header	0.363	0.800	0.120	0.190	0.05	0.05	0.0024	0.01	---	---
Pyrene	129-00-0	Refer to Column Header	0.341	0.800	0.0916	0.190	0.05	0.05	0.0017	0.01	---	---
C1-pyrene/fluoranthenes	---	Refer to Column Header	---	---	---	---	0.1	0.1	0.01	0.01	---	---
Benzo(a)anthracene	56-55-3	Refer to Column Header	0.0453	0.160	0.119	0.190	0.05	0.05	0.0015	0.01	---	---
Chrysene	218-01-9	Refer to Column Header	0.0545	0.160	0.0837	0.190	0.05	0.05	0.00022	0.01	---	---
C1-benzo(a)anthracene/chrysenes	---	Refer to Column Header	---	---	---	---	0.100	0.100	0.01	0.01	---	---
C2-benzo(a)anthracene/chrysenes	---	Refer to Column Header	---	---	---	---	0.100	0.100	0.01	0.01	---	---
C3-benzo(a)anthracene/chrysenes	---	Refer to Column Header	---	---	---	---	0.100	0.100	0.01	0.01	---	---
C4-benzo(a)anthracene/chrysenes	---	Refer to Column Header	---	---	---	---	0.100	0.100	0.01	0.01	---	---
Benzo(b)fluoranthene	205-99-2	Refer to Column Header	0.0645	0.160	0.0658	0.190	0.05	0.05	0.0015	0.01	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits	
			Chicago		Pittsburgh		Nashville		Knoxville		Canton	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Benzo(k)fluoranthene	207-08-9	Refer to Column Header	0.0512	0.160	0.185	0.190	0.05	0.05	0.001	0.01	---	---
Benzo(a)pyrene	50-32-8	Refer to Column Header	0.0791	0.160	0.0889	0.190	0.05	0.05	0.0004	0.01	---	---
Perylene	198-55-0	Refer to Column Header	---	---	---	---	0.05	0.05	0.00081	0.01	---	---
Benzo(e)pyrene	192-97-2	Refer to Column Header	---	---	0.0567	1.0	0.05	0.05	0.0014	0.01	---	---
Indeno(1,2,3-cd)pyrene	193-39-5	Refer to Column Header	0.0598	0.160	0.0771	0.190	0.05	0.05	0.001	0.01	---	---
Dibenzo(a,h)anthracene	53-70-3	Refer to Column Header	0.0406	0.240	0.0776	0.190	0.05	0.05	0.00078	0.01	---	---
Benzo(g,h,i)perylene	191-24-2	Refer to Column Header	0.300	0.800	0.0815	0.190	0.05	0.05	0.00051	0.01	---	---
1-Methylnaphthalene	90-12-0	Refer to Column Header	0.241	1.60	0.0873	0.190	0.05	0.05	0.0041	0.01	---	---
2-Methylnaphthalene	91-57-6	Refer to Column Header	0.0521	1.60	0.115	0.190	0.05	0.05	0.0083	0.01	---	---
Bis(2-Ethylhexyl)Phthalate	117-81-7	Refer to Column Header	1.37	8.0	1.92	2.0	---	---	---	---	---	---
n-Nitrosodiphenylamine	86-30-6	Refer to Column Header	0.296	1.6	0.106	1.0	---	---	---	---	---	---
Carbazole	86-74-8	Refer to Column Header	0.283	4.00	0.063	0.19	---	---	---	---	---	---
Dibenzofuran	132-64-9	Refer to Column Header	0.210	1.60	0.116	1.0	0.0500	0.100	0.02	0.2	---	---
Phenols												
2,4-Dichlorophenol	120-83-2	SW846-8270C/D	0.0579	0.190	0.0579	0.19	---	---	---	---	---	---
2,4-Dimethylphenol	105-67-9	SW846-8270C/D	0.0762	1.00	0.0762	1	---	---	---	---	---	---
2-Methylphenol (o-cresol)	95-48-7	SW846-8270C/D	0.235	1.00	0.235	1	---	---	---	---	---	---
3&4-Methylphenol (m, p-cresol)	106-44-5	SW846-8270C/D	0.242	1.00	0.242	1	---	---	---	---	---	---
4-Methylphenol	106-44-5	SW846-8270C/D	0.242	1.00	0.242	1	---	---	---	---	---	---
Phenol	108-95-2	SW846-8270C/D	0.299	1.00	0.299	1	---	---	---	---	---	---
Phenolics	Multiple	SW846 9066	4.1	5.0	---	---	---	---	---	---	---	---
Pesticides												
Aldrin	309-00-2	SW846 8081B	0.0053	0.04	---	---	---	---	---	---	---	---
α-BHC	319-84-6	SW846 8081B	0.0026	0.04	---	---	---	---	---	---	---	---
β-BHC	319-85-7	SW846 8081B	0.0102	0.04	---	---	---	---	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	0.0056	0.04	---	---	---	---	---	---	---	---
δ-BHC	319-86-8	SW846 8081B	0.0103	0.04	---	---	---	---	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081B	0.0044	0.04	---	---	---	---	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081B	0.0072	0.04	---	---	---	---	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	0.0376	0.08	---	---	---	---	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	---	---	---	---	---	---	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081B	0.0133	0.04	---	---	---	---	---	---	---	---
4,4'-DDE	72-55-9	SW846 8081B	0.0038	0.04	---	---	---	---	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081B	0.0032	0.04	---	---	---	---	---	---	---	---
Diallate	2303-16-4	SW846 8081B	---	---	---	---	---	---	---	---	---	---
Dieldrin	60-57-1	SW846 8081B	0.0129	0.04	---	---	---	---	---	---	---	---
Endosulfan I	959-98-8	SW846 8081B	0.0041	0.04	---	---	---	---	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081B	0.0028	0.04	---	---	---	---	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	0.0117	0.04	---	---	---	---	---	---	---	---
Endrin	72-20-8	SW846 8081B	0.0142	0.04	---	---	---	---	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	0.0082	0.04	---	---	---	---	---	---	---	---
Endrin ketone	53494-70-5	SW846 8081B	0.017	0.04	---	---	---	---	---	---	---	---
Heptachlor	76-44-8	SW846 8081B	0.0135	0.04	---	---	---	---	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	0.0138	0.04	---	---	---	---	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	---	---	---	---	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	---	---	---	---	---	---	---	---	---	---
Isodrin	465-73-6	SW846 8081B	0.02	0.04	---	---	---	---	---	---	---	---
Methoxychlor	72-43-5	SW846 8081B	0.023	0.08	---	---	---	---	---	---	---	---
Toxaphene	8001-35-2	SW846 8081B	0.2	0.4								
PCBs												
Aroclor 1016	12674-11-2	SW846 8082A	0.067	0.4	---	---	---	---	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	0.2	0.4	---	---	---	---	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	0.2	0.4	---	---	---	---	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	0.2	0.4	---	---	---	---	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	0.2	0.4	---	---	---	---	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	0.2	0.4	---	---	---	---	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits	
			Chicago		Pittsburgh		Nashville		Knoxville		Canton	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Aroclor 1260	11096-82-5	SW846 8082A	0.07	0.4	---	---	---	---	---	---	---	---
PCB Congeners			ng/L	ng/L								
2-Chlorobiphenyl	2051-60-7	SW846 8082A	---	---	---	---	---	---	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	---	---	---	---	---	---	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	0.254	1.00	---	---	---	---	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	---	---	---	---	---	---	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	0.27	1	---	---	---	---	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	0.466	1	---	---	---	---	---	---	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	0.23	1	---	---	---	---	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	0.249	1	---	---	---	---	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	0.275	1	---	---	---	---	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	0.227	1	---	---	---	---	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	---	---	---	---	---	---	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	---	---	---	---	---	---	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	0.277	1	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	0.336	1	---	---	---	---	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	0.308	1	---	---	---	---	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	0.836	1	---	---	---	---	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	0.308	1	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	0.341	1	---	---	---	---	---	---	---	---
Inorganics												
Aluminum	7429-90-5	SW846-6020A	24.6	100.0	---	---	---	---	---	---	---	---
Antimony	7440-36-0	SW846-6020A	1.32	3.00	---	---	---	---	---	---	---	---
Arsenic, total	7440-38-2	SW846-6020A	0.23	1.00	---	---	---	---	---	---	---	---
Barium, total	7440-39-3	SW846-6020A	0.73	2.50	---	---	---	---	---	---	---	---
Cadmium	7440-43-9	SW846-6020A	0.167	0.50	---	---	---	---	---	---	---	---
Chromium III	---	SM 3500_CR3_B	0.01	0.01	---	---	---	---	---	---	---	---
Chromium VI	18540-29-9	SM 3500_CR3_B	0.003	0.01	---	---	---	---	---	---	---	---
Chromium, total	7440-47-3	SW846-6020A	1.14	5.00	---	---	---	---	---	---	---	---
Copper, total	7440-50-8	SW846-6020A	0.497	2.00	---	---	---	---	---	---	---	---
Cyanide, total	57-12-5	SW846-9010B/9010C	3.45	10	---	---	---	---	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	---	---	0.36	2	---	---	---	---	---	---
Cyanide, amenable	57-12-5	SW846 9010C/9010C	3.45	10	---	---	---	---	---	---	---	---
Cyanide, dissociable	57-12-5	SM 4500CN	3.42	10	---	---	---	---	---	---	---	---
Iron, dissolved	7439-89-6	SW846-6020A	46.7	100.0	---	---	---	---	---	---	---	---
Iron, total	7439-89-6	SW846-6020A	46.7	100.0	---	---	---	---	---	---	---	---
Lead	7439-92-1	SW846-6020A	0.186	0.50	---	---	---	---	---	---	---	---
Manganese, total	7439-96-5	SW846-6020A	0.791	2.50	---	---	---	---	---	---	---	---
Mercury	7439-97-6	SW846-7471A/7471B	0.0984	0.20	---	---	---	---	---	---	---	---
Nickel, total	7440-2-0	SW846-6020A	0.625	2.00	---	---	---	---	---	---	---	---
Selenium, total	7782-49-2	SW846-6020A	0.982	2.50	---	---	---	---	---	---	---	---
Silver, total	7440-22-4	SW846-6020A	0.115	0.50	---	---	---	---	---	---	---	---
Thallium, total	7440-28-0	SW846-6020A	0.57	2.00	---	---	---	---	---	---	---	---
Vanadium	7440-62-2	SW846-6020A	2.15	5.00	---	---	---	---	---	---	---	---
Zinc, total	7440-66-6	SW846-6020A	6.92	20.00	---	---	---	---	---	---	---	---
Other												
Alkalinity as CaCO ₃	3812-32-6	SM 2320B	3740	5000	---	---	---	---	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	100	200	---	---	---	---	---	---	---	---
Biochemical Oxygen Demand (BOD)	---	SM 5210B	2000	2000	---	---	---	---	---	---	---	---
Chloride	16887-00-6	SW846 9056A	170	200	---	---	---	---	---	---	---	---
Chemical Oxygen Demand (COD)	---	SM5220C	6040	10000	---	---	---	---	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	56.3	100	---	---	---	---	---	---	---	---
Hardness	---	SW846 6010/SM2340B Calc.	660	1320	---	---	---	---	---	---	---	---
Methane	74-82.8	RSK-175	---	---	---	---	---	---	---	---	0.08	0.50
Nitrate	14797-55-8	SW846 9056A	68.0	200	---	---	---	---	---	---	---	---
Nitrogen, Nitrate & Nitrite	14797-55-8 &14797-65-0	EPA Method 353.2	41.1	100	---	---	---	---	---	---	---	---

Table 1B

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits	
			Chicago		Pittsburgh		Nashville		Knoxville		Canton	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Oil & Grease	---	EPA Method 1664A	1320	5000	---	---	---	---	---	---	---	---
pH	---	EPA Method 150.1/ SM 9040/ SM 9045	0.2 SU	0.2 SU	---	---	---	---	---	---	---	---
Phosphate	14265-44-2	SM 4500_P_E	73	153	---	---	---	---	---	---	---	---
Residue, Non-filterable Total Suspended Solids (TSS)	---	SM 2540D	1930	5000	---	---	---	---	---	---	---	---
Sulfate	14808-79-8	SW846 9056A	95	200	---	---	---	---	---	---	---	---
Sulfur	7704-34-9	SW846 6010	---	---	---	---	100	250	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151	0.0898	0.50	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060	470	1000	---	---	---	---	---	---	---	---

O: SLMC: SSWF: SLM

Notes:

aka = also known as

CAS = Chemical Abstracts Service

CaCO₃ = Calcium Carbonate

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

GC= gas chromatography

LL = low level

MDL = Method Detection Limit

MGP = Manufactured Gas Plant

MS = mass spectrometry

OIA 1667 = OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCBs = polychlorinated biphenyls

RAF = Multi-Site Risk Assessment Framework

RCRA = Resource Conservation and Recovery Act

RL = Reporting Limit

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP = Standard operating procedure

SU = Standard Unit (pH is dimensionless)

SVOC = semivolatile organic compound

SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition

TPH = Total Petroleum Hydrocarbons

USEPA or EPA = United States Environmental Protection Agency

VOA = volatile organic analyte/analysis

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

µg/L = micrograms per liter

** Note from lab: C1-naphthalenes includes 1-methylnapthalene and 2-methylnapthalene, see SOP for additional table entries for these analytes.

--- = Lab does not offer analysis, analysis was not requested, no data

^A Prior to any project-specific field data collection, the selected lab’s ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for surface water and groundwater. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 1C. Multi-Site Program Water Matrix - Lowest Achievable Limits - Eurofins Only
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Volatile Organic Compounds								
Benzene	71-43-2	SW846-8260B/C	0.5	1	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B/C	0.5	1	---	---	---	---
Toluene	108-88-3	SW846-8260B/C	0.5	1	---	---	---	---
Xylenes, total	1330-20-7	SW846-8260B/C	0.5	1	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B/C	1	5	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B/C	1	5	---	---	---	---
Methyl-Tert-Butyl Ether	1634-04-4	SW846-8260B/C	0.5	1	---	---	---	---
1,1,2,2-Tetrachloroethane	79-34-5	SW846-8260B/C	0.5	1	---	---	---	---
Tetrachloroethene	127-18-4	SW846-8260B/C	0.5	1	---	---	---	---
Trichloroethene	79-01-6	SW846-8260B/C	0.5	1	---	---	---	---
Vinyl Chloride	75-01-4	SW846-8260B/C	0.5	1	---	---	---	---
Chloroform	67-66-3	SW846-8260B/C	0.5	1	---	---	---	---
Isopropylbenzene (aka Cumene)	98-82-8	SW846-8260B/C	1.0	5	---	---	---	---
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	---	---	---	---	---	---
Semivolatile Organic Compounds					8270C SIM	8270C SIM	8270C/D	8270C/D
Naphthalene	91-20-3	SW846-8270C/D/SIM PAH	0.02	0.05	0.030	0.060	0.1	0.5
Nitrobenzene	98-95-3	SW846-8270C/D	0.5	1	---	---	---	---
C1-naphthalenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C2-naphthalenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C3-naphthalenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C4-naphthalenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Acenaphthylene	208-96-8	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Acenaphthene	83-32-9	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Fluorene	86-73-7	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
C1-fluorenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C2-fluorenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C3-fluorenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Phenanthrene	85-01-8	SW846-8270C/D/SIM PAH	0.02	0.05	0.030	0.060	0.1	0.5
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C1-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C2-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C3-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C4-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Fluoranthene	206-44-0	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Pyrene	129-00-0	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
C1-pyrene/fluoranthenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Benzo(a)anthracene	56-55-3	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Chrysene	218-01-9	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
C1-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C2-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
C3-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
C4-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Benzo(b)fluoranthene	205-99-2	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Benzo(k)fluoranthene	207-08-9	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Benzo(a)pyrene	50-32-8	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Perylene	198-55-0	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Benzo(e)pyrene	192-97-2	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Indeno(1,2,3-cd)pyrene	193-39-5	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Dibenzo(a,h)anthracene	53-70-3	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
Benzo(g,h,i)perylene	191-24-2	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
1-Methylnaphthalene	90-12-0	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
2-Methylnaphthalene	91-57-6	SW846-8270C/D/SIM PAH	0.02	0.05	0.010	0.050	0.1	0.5
1,2-Dichloroethene	540-59-0	SW846-8270C/D/SIM PAH	0.5	1	---	---	---	---
cis-1,2-Dichloroethene	156-59-2	SW846-8270C/D/SIM PAH	0.5	1	---	---	---	---
trans-1,2-Dichloroethene	156-60-5	SW846-8270C/D/SIM PAH	0.5	1	---	---	---	---
Bis(2-Ethylhexyl)Phthalate	117-81-7	SW846-8270C/D/SIM PAH	2	5	---	---	---	---
p-Isopropyltoluene	99-87-6	SW846-8270C/D/SIM PAH	1	5	---	---	---	---
Dichloromethane	75-09-2	SW846-8270C/D/SIM PAH	2	4	---	---	---	---
n-Nitrosodiphenylamine	86-30-6	SW846-8270C/D/SIM PAH	0.5	1	---	---	---	---
Carbazole	86-74-8	SW846-8270C/D/SIM PAH	0.5	1	---	---	---	---
Dibenzofuran	132-64-9	SW846-8270C/D/SIM PAH	0.02	0.05	---	---	---	---
Phenols								
2,4-Dichlorophenol	120-83-2	SW846-8270C/D	0.5	1	---	---	---	---
2,4-Dimethylphenol	105-67-9	SW846-8270C/D	0.5	1	---	---	---	---
2-Methylphenol (o-cresol)	95-48-7	SW846-8270C/D	0.5	1	---	---	---	---
3&4-Methylphenol (m, p-cresol)	106-44-5	SW846-8270C/D	0.5	1	---	---	---	---
4-Methylphenol	106-44-5	SW846-8270C/D	0.5	1	---	---	---	---
Phenol	108-95-2	SW846-8270C/D	0.5	1	---	---	---	---
Phenolics	Multiple	SW846 9066	0.01	0.02	---	---	---	---
Pesticides								
Aldrin	309-00-2	SW846 8081A/B	0.002	0.010	---	---	---	---
α-BHC	319-84-6	SW846 8081A/B	0.003	0.010	---	---	---	---
β-BHC	319-85-7	SW846 8081A/B	0.003	0.010	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081A/B	0.002	0.010	---	---	---	---
δ-BHC	319-86-8	SW846 8081A/B	0.002	0.010	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081A/B	0.003	0.010	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081A/B	0.007	0.020	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081A/B	0.16	0.5	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081A/B	3	10	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081A/B	2	5	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081A/B	0.005	0.020	---	---	---	---
4,4'-DDE	72-55-9	SW846 8081A/B	0.005	0.020	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081A/B	0.005	0.020	---	---	---	---
Diallate	2303-16-4	SW846 8081A/B	1	5	---	---	---	---
Dieldrin	60-57-1	SW846 8081A/B	0.005	0.020	---	---	---	---
Endosulfan I	959-98-8	SW846 8081A/B	0.004	0.010	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081A/B	0.015	0.030	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081A/B	0.005	0.020	---	---	---	---
Endrin	72-20-8	SW846 8081A/B	0.005	0.020	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081A/B	0.020	0.010	---	---	---	---

Table 1C

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Endrin ketone	53494-70-5	SW846 8081A/B	0.005	0.020	---	---	---	---
Heptachlor	76-44-8	SW846 8081A/B	0.002	0.010	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081A/B	0.002	0.010	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081A/B	0.01	0.003	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081A/B	5	15	---	---	---	---
Isodrin	465-73-6	SW846 8081A/B	0.5	1	---	---	---	---
Methoxychlor	72-43-5	SW846 8081A/B	0.03	0.1	---	---	---	---
Toxaphene	8001-35-2	SW846 8081A/B	0.3	1.0	---	---	---	---
PCBs								
Aroclor 1016	12674-11-2	SW846 8082A	0.1	0.5	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	0.1	0.5	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	0.2	0.5	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	0.1	0.5	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	0.1	0.5	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	0.1	0.5	---	---	---	---
Aroclor 1260	11096-82-5	SW846 8082A	0.15	0.5	---	---	---	---
PCB Congeners								
2-Chlorobiphenyl	2051-60-7	SW846 8082A	10	20	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	8	20	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	16	50	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	18	50	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	40	100	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	15	50	---	---	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	17	50	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	74	200	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	47	200	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	39	100	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	63	200	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	17	50	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	46	100	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	30	100	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	12	50	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	30	100	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	28	100	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	17	50	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	16	50	---	---	---	---
Inorganics			6010B/7470A or 200.7/245.1	6010B/7470A or 200.7/245.1	6010C/D and 7470A	6010C/D and 7470A		
Aluminum	7429-90-5	SW846-6020A/ 6010	89.4	200	89.4	400	---	---
Antimony	7440-36-0	SW846-6020A/ 6010	8.7	20	8.7	40	---	---
Arsenic, total	7440-38-2	SW846-6020A/ 6010	9.6	20	9.6	40	---	---
Barium, total	7440-39-3	SW846-6020A/ 6010	0.85	5	0.85	10	---	---
Cadmium	7440-43-9	SW846-6020A/ 6010	1.8	5	1.8	10	---	---
Chromium III	---	SW846-6020A/ 6011	1.8	15	---	---	---	---
Chromium VI	18540-29-9	SW846-6020A/ 6012	10	30	---	---	---	---
Chromium, total	7440-47-3	SW846-6020A/ 6010	3.3	15	3.3	30	---	---
Copper, total	7440-50-8	SW846-6020A/ 6010	4	10	4	20	---	---

Table 1C

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Cyanide, total	57-12-5	SW846-9012A/B	5	10	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	2	6	---	---	---	---
Cyanide, amenable	57-12-5	SW846 9012B/9014	5	10	---	---	---	---
Cyanide, dissociable	57-12-5	SM 4500CN	5	10	---	---	---	---
Iron, dissolved	7439-89-6	SW846-6020A/ 6010	---	---	---	---	---	---
Iron, total	7439-89-6	SW846-6020A/ 6011	80.5	200	80.5	400	---	---
Lead	7439-92-1	SW846-6020A/ 6010	6	15	6	30	---	---
Manganese, total	7439-96-5	SW846-6020A/ 6010	1.6	5	1.6	10	---	---
Mercury	7439-97-6	SW846-7471B	0.05	0.2	0.05	0.2	---	---
Nickel, total	7440-2-0	SW846-6020A/ 6010	4	10	4	20	---	---
Selenium, total	7782-49-2	SW846-6020A/ 6010	9.3	20	9.3	40	---	---
Silver, total	7440-22-4	SW846-6020A/ 6010	2.4	5	2.4	10	---	---
Thallium, total	7440-28-0	SW846-6020A/ 6011	13.7	30	13.7	60	---	---
Vanadium	7440-62-2	SW846-6020A/ 6010	1.6	5	1.6	10	---	---
Zinc, total	7440-66-6	SW846-6020A/ 6010	6.5	20	6.5	40	---	---
Other								
Alkalinity as CaCO ₃	3812-32-6	SM 2320B	1700	5000	---	---	---	---
Ammonia	7644-41-7	EPA 350.1	50	100	---	---	---	---
Biochemical Oxygen Demand (BOD)	---	SM 5210B	2000	2000	---	---	---	---
Chloride	16887-00-6	SM 4500-CI E	600	2000	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	12800	50000	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	30	100	---	---	---	---
Hardness	---	SW846 6010	3000	10000	---	---	---	---
Methane	74-82.8	RSK-175	3000	5000	---	---	---	---
Nitrate	14797-55-8	EPA Method 353.2	40	100	---	---	---	---
Nitrogen, Nitrate & Nitrite	14797-55-8 &14797-65-0	EPA Method 353.2	10	100	---	---	---	---
Oil & Grease	---	EPA Method 1664A	1400	5000	---	---	---	---
pH	---	EPA Method 150.1/ SM 9040/ SM 9045	10 SU	10 SU	---	---	---	---
Phosphate	14265-44-2	EPA Method 365.1	50	100	---	---	---	---
Residue, Non-filterable Total Suspended Solids (TSS)	---	SM 2540D	1000	3000	---	---	---	---
Sulfate	14808-79-8	SM 4500 SO4 E	2500	5000	---	---	---	---
Sulfur	7704-34-9	SW846 6010	174	500	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151	0.027	0.05	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060	500	1000	---	---	---	---
Alternate Methods Provided								
Chromium VI	18540-29-9	SW846 7196A	10	30	---	---	---	---
Hardness	---	SM 2340C	3000	10000	---	---	---	---
Sulfate	7704-34-9	EPA Method 375.4	2500	5000	---	---	---	---
Chlorobenzilate	510-15-6	Method SW-846 8270	3	10	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	Method SW-846 8260	2	5	---	---	---	---
Diallate	2303-16-4	Method SW-846 8270	1	5	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	Method SW-846 8270	5	15	---	---	---	---
Isodrin	465-73-6	Method SW-846 8270	0.5	1	---	---	---	---
2-Chlorobiphenyl	2051-60-7	EPA Method 1668A or C	10	20	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	EPA Method 1668A or C	8	20	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	EPA Method 1668A or C	16	50	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	EPA Method 1668A or C	18	50	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	EPA Method 1668A or C	40	100	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	EPA Method 1668A or C	15	50	---	---	---	---

Table 1C

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	EPA Method 1668A or C	17	50	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	EPA Method 1668A or C	74	200	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	EPA Method 1668A or C	47	200	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	EPA Method 1668A or C	39	100	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	EPA Method 1668A or C	63	200	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	EPA Method 1668A or C	17	50	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	EPA Method 1668A or C	46	100	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	EPA Method 1668A or C	30	100	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	EPA Method 1668A or C	12	50	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	EPA Method 1668A or C	30	100	---	---	---	---
2,2',3,4,4',5,6-Heptachlorobiphenyl	52663-69-1	EPA Method 1668A or C	28	100	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	EPA Method 1668A or C	17	50	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	EPA Method 1668A or C	16	50	---	---	---	---

O: SLM C: SSW F: SLM

Notes:
aka = also known as
CAS = Chemical Abstracts Service
CaCO₃ = Calcium Carbonate
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136.
MDL = Method Detection Limit
MGP = Manufactured Gas Plant
OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
PAH = polycyclic aromatic hydrocarbon
PCBs = polychlorinated biphenyls
RAF = Multi-Site Risk Assessment Framework
RCRA = Resource Conservation and Recovery Act
RL = Reporting Limit
RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP = Standard operating procedure
SU= Standard Unit (pH is dimensionless)
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
TPH = Total Petroleum Hydrocarbons
USEPA or EPA = United States Environmental Protection Agency
VOA = volatile organic analyte/analysis
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
µg/L = micrograms per liter
--- = Lab does not offer analysis, analysis was not requested, no data
Bold Underline Text = Lab will provide an alternate method than what is identified in method column

^A Prior to any project-specific field data collection, the selected lab’s ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for surface water and groundwater. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 1D. Multi-Site Program Water Matrix - Lowest Achievable Limits - Pace Analytical Only
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
			8260	8260	8021	8021				
Volatile Organic Compounds										
Benzene	71-43-2	SW846-8260B/ 8021B	0.396	1	0.396	1.00	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B/ 8021B	0.393	1	0.393	1.00	---	---	---	---
Toluene	108-88-3	SW846-8260B/ 8021B	0.388	1	0.388	1.00	---	---	---	---
Xylenes, total	1330-20-7	SW846-8260B/ 8021B	0.449	3	1.247	3.00	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B/ 8021B	0.416	1	0.416	1.00	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B/ 8021B	0.418	1	0.418	1.00	---	---	---	---
Methyl-Tert-Butyl Ether	1634-04-4	SW846-8260B/ 8021B	0.485	1	0.485	1.00	---	---	---	---
1,1,2,2-Tetrachloroethane	79-34-5	SW846-8260B/ 8021B	---	---	---	---	---	---	0.22	1
Tetrachloroethene	127-18-4	SW846-8260B/ 8021B	---	---	---	---	---	---	0.26	1
Trichloroethene	79-01-6	SW846-8260B/ 8021B	---	---	---	---	---	---	0.26	1
Vinyl Chloride	75-01-4	SW846-8260B/ 8021B	---	---	---	---	---	---	0.27	1
Chloroform	67-66-3	SW846-8260B/ 8021B	---	---	---	---	---	---	0.2	1
1,2-Dichloroethene	540-59-0	SW846-8270C/D/SIM PAH	---	---	---	---	---	---	0.51	2
cis-1,2-Dichloroethene	156-59-2	SW846-8270C/D/SIM PAH	---	---	---	---	---	---	0.25	1
trans-1,2-Dichloroethene	156-60-5	SW846-8270C/D/SIM PAH	---	---	---	---	---	---	0.26	1
p-Isopropyltoluene	99-87-6	SW846-8260B	---	---	---	---	---	---	0.21	1
Dichloromethane	75-09-2	SW846-8260B	---	---	---	---	---	---	0.24	1
Isopropylbenzene (aka Cumene)	98-82-8	SW846-8260B/ 8021B	---	---	---	---	---	---	0.1	1
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	15.5	51.6	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	29.592	50	---	---	---	---	---	---
Semivolatile Organic Compounds			8270 SIM	8270 SIM	8270	8270	Alkylated	Alkylated		
Naphthalene	91-20-3	SW846-8270C/D/SIM PAH	0.01833	0.0916	1.8986	6.3288	0.01200	0.04	0.017	0.06
Nitrobenzene	98-95-3	SW846-8270C/D	---	---	1.4501	4.8337	---	---	0.058	0.5
C1-naphthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C2-napthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C3-napthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C4-napthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
Acenaphthylene	208-96-8	SW846-8270C/D/SIM PAH	0.00498	0.0249	1.0617	3.5391	0.00910	0.04	0.0021	0.06
Acenaphthene	83-32-9	SW846-8270C/D/SIM PAH	0.00607	0.0303	1.3394	4.4645	0.01000	0.04	0.013	0.06
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.01045	0.0523	1.8059	6.0197	0.01000	0.04	---	---
Fluorene	86-73-7	SW846-8270C/D/SIM PAH	0.00797	0.0399	0.7499	2.4998	0.00880	0.04	0.012	0.05
C1-fluorenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C2-fluorenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C3-fluorenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
Phenanthrene	85-01-8	SW846-8270C/D/SIM PAH	0.01379	0.0689	1.8211	6.0705	0.00790	0.04	0.016	0.05
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.01045	0.0523	1.8059	6.0197	0.01000	0.04	---	---
C1-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C2-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C3-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C4-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
Fluoranthene	206-44-0	SW846-8270C/D/SIM PAH	0.01067	0.0533	0.5634	1.8780	0.01100	0.04	0.013	0.05
Pyrene	129-00-0	SW846-8270C/D/SIM PAH	0.00765	0.0383	1.3469	4.4897	0.01100	0.04	0.0058	0.05
C1-pyrene/fluoranthenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
Benzo(a)anthracene	56-55-3	SW846-8270C/D/SIM PAH	0.00755	0.0378	0.5349	1.7831	0.01300	0.04	0.0059	0.05
Chrysene	218-01-9	SW846-8270C/D/SIM PAH	0.01305	0.0652	1.7395	5.7984	0.01400	0.04	0.0029	0.05
C1-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C2-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C3-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
C4-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	0.02000	0.04	---	---
Benzo(b)fluoranthene	205-99-2	SW846-8270C/D/SIM PAH	0.00574	0.0287	0.6540	2.1801	0.01600	0.04	0.0053	0.05
Benzo(k)fluoranthene	207-08-9	SW846-8270C/D/SIM PAH	0.00755	0.0377	1.0027	3.3425	0.02000	0.04	0.0043	0.05
Benzo(a)pyrene	50-32-8	SW846-8270C/D/SIM PAH	0.01053	0.0526	1.8826	6.2755	0.01300	0.04	0.0029	0.05
Perylene	198-55-0	SW846-8270C/D/SIM PAH	---	---	---	---	0.01300	0.04	---	---
Benzo(e)pyrene	192-97-2	SW846-8270C/D/SIM PAH	---	---	---	---	0.00980	0.04	---	---
Indeno(1,2,3-cd)pyrene	193-39-5	SW846-8270C/D/SIM PAH	0.01764	0.0882	1.4979	4.9930	0.02000	0.04	0.01	0.05
Dibenzo(a,h)anthracene	53-70-3	SW846-8270C/D/SIM PAH	0.01002	0.0501	1.3213	4.4045	0.01700	0.04	0.01	0.05
Benzo(g,h,i)perylene	191-24-2	SW846-8270C/D/SIM PAH	0.00678	0.0339	0.8107	2.7024	0.01900	0.04	0.0079	0.05

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
1-Methylnaphthalene	90-12-0	SW846-8270C/D/SIM PAH	0.0059	0.0295	1.6681	5.5605	0.00890	0.04	0.0145	0.06
2-Methylnaphthalene	91-57-6	SW846-8270C/D/SIM PAH	0.0049	0.0245	1.5142	5.0472	0.01100	0.04	0.013	0.06
Bis(2-Ethylhexyl)Phthalate	117-81-7	SW846-8270C/D/SIM PAH	---	---	0.6932	2.3108	---	---	0.11	0.5
p-Isopropyltoluene	99-87-6	SW846-8270C/D/SIM PAH	---	---	---	---	---	---	0.21	1
Dichloromethane	75-09-2	SW846-8270C/D/SIM PAH	---	---	---	---	---	---	0.24	1
n-Nitrosodiphenylamine	86-30-6	SW846-8270C/D/SIM PAH	---	---	3.5279	11.7595	---	---	0.1	0.5
Carbazole	86-74-8	SW846-8270C/D/SIM PAH	---	---	0.7495	2.4984	---	---	0.07	0.5
Dibenzofuran	132-64-9	SW846-8270C/D/SIM PAH	---	---	0.7685	2.5618	---	---	0.041	0.5
Phenols										
2,4-Dichlorophenol	120-83-2	SW846-8270C/D	1.3667	4.5555	---	---	---	---	0.092	0.5
2,4-Dimethylphenol	105-67-9	SW846-8270C/D	1.2651	4.2168	---	---	---	---	0.17	1
2-Methylphenol (o-cresol)	95-48-7	SW846-8270C/D	0.8681	2.8938	---	---	---	---	0.048	0.5
3&4-Methylphenol (m, p-cresol)	106-44-5	SW846-8270C/D	1.5618	5.2060	---	---	---	---	0.057	0.5
4-Methylphenol	106-44-5	SW846-8270C/D	---	---	---	---	---	---	0.057	0.5
Phenol	108-95-2	SW846-8270C/D	0.5995	1.9984	---	---	---	---	0.034	0.5
Phenolics	Multiple	SW846 9066	3.38	10.00	---	---	---	---	---	---
Pesticides										
Aldrin	309-00-2	SW846 8081B	0.0074	0.025	---	---	---	---	---	---
α-BHC	319-84-6	SW846 8081B	0.0079	0.026	---	---	---	---	---	---
β-BHC	319-85-7	SW846 8081B	0.0081	0.027	---	---	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	0.0063	0.021	---	---	---	---	---	---
δ-BHC	319-86-8	SW846 8081B	0.0118	0.039	---	---	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081B	0.0290	0.097	---	---	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081B	0.0068	0.023	---	---	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	0.2182	0.727	---	---	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081B	---	---	---	---	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	---	---	---	---	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081B	0.0141	0.047	---	---	---	---	---	---
4,4'-DDE	72-55-9	SW846 8081B	0.0184	0.061	---	---	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081B	0.0142	0.047	---	---	---	---	---	---
Diallate	2303-16-4	SW846 8081B	---	---	---	---	---	---	---	---
Dieldrin	60-57-1	SW846 8081B	0.0134	0.045	---	---	---	---	---	---
Endosulfan I	959-98-8	SW846 8081B	0.0097	0.032	---	---	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081B	0.0240	0.080	---	---	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	0.0149	0.050	---	---	---	---	---	---
Endrin	72-20-8	SW846 8081B	0.0157	0.052	---	---	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	0.0155	0.052	---	---	---	---	---	---
Endrin ketone	53494-70-5	SW846 8081B	0.0154	0.051	---	---	---	---	---	---
Heptachlor	76-44-8	SW846 8081B	0.0065	0.022	---	---	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	0.0130	0.043	---	---	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	0.0117	0.039	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	---	---	---	---	---	---	---	---
Isodrin	465-73-6	SW846 8081B	---	---	---	---	---	---	---	---
Methoxychlor	72-43-5	SW846 8081B	0.0812	0.271	---	---	---	---	---	---
Toxaphene	8001-35-2	SW846 8081B	1.5000	3.00	---	---	---	---	---	---
PCBs										
Aroclor 1016	12674-11-2	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
Aroclor 1260	11096-82-5	SW846 8082A	0.25000	0.5	---	---	---	---	---	---
PCB Congeners										
2-Chlorobiphenyl	2051-60-7	SW846 8082A	---	---	---	---	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	---	---	---	---	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	---	---	---	---	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	---	---	---	---	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	---	---	---	---	---	---	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	---	---	---	---	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	---	---	---	---	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	---	---	---	---	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	---	---	---	---	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	---	---	---	---	---	---	---	---
Inorganics			SW846-6020	SW846-6020	SW846-6010	SW846-6010				
Aluminum	7429-90-5	SW846-6020A/ 6010	58.700	250.00	55.5	500.00	---	---	---	---
Antimony	7440-36-0	SW846-6020A/ 6010	0.150	1.00	7.5797	25.00	---	---	---	---
Arsenic, total	7440-38-2	SW846-6020A/ 6010	0.279	1.00	8.345	25.00	---	---	---	---
Barium, total	7440-39-3	SW846-6020A/ 6010	0.341	1.14	1.5	5.00	---	---	---	---
Cadmium	7440-43-9	SW846-6020A/ 6010	0.081	1.00	1.33	5.00	---	---	---	---
Chromium III	---	SW846-6020A/ 6011	---	---	---	---	---	---	---	---
Chromium VI	18540-29-9	SW846-6020A/ 6012	5.1	20	---	---	---	---	---	---
Chromium, total	7440-47-3	SW846-6020A/ 6010	1.0199	3.40	2.546	10.00	---	---	---	---
Copper, total	7440-50-8	SW846-6020A/ 6010	1.093	3.65	6.28	20.00	---	---	---	---
Cyanide, total	57-12-5	SW846-9012A	6.79	23	---	---	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	0.329	2	---	---	---	---	---	---
Cyanide, amenable	57-12-5	SW846 9012B/9014	1	5	---	---	---	---	---	---
Cyanide, dissociable	57-12-5	SM 4500CN	1.8	5	---	---	---	---	---	---
Iron, dissolved	7439-89-6	SW846-6020A/ 6010	110.551	368.50	33.99	100.00	---	---	---	---
Iron, total	7439-89-6	SW846-6020A/ 6011	110.551	368.5	33.99	100.00	---	---	---	---
Lead	7439-92-1	SW846-6020A/ 6010	0.195	1.00	4.33	13.00	---	---	---	---
Manganese, total	7439-96-5	SW846-6020A/ 6010	2.700	9.00	1.8302	5.50	---	---	---	---
Mercury	7439-97-6	SW846-7471B	0.179	0.597	---	---	---	---	---	---
Nickel, total	7440-2-0	SW846-6020A/ 6010	0.402	1.34	2.622	10.00	---	---	---	---
Selenium, total	7782-49-2	SW846-6020A/ 6010	0.317	1.06	16.559	50.00	---	---	---	---
Silver, total	7440-22-4	SW846-6020A/ 6010	0.101	0.50	3.316	10.00	---	---	---	---
Thallium, total	7440-28-0	SW846-6020A/ 6011	0.140	1.00	7.38	40.00	---	---	---	---
Vanadium	7440-62-2	SW846-6020A/ 6010	0.316	1.05	2.23	10.00	---	---	---	---
Zinc, total	7440-66-6	SW846-6020A/ 6010	4.600	15.33	9.330	40.00	---	---	---	---
Other					EPA 310.2 and SM 5310C	EPA 310.2 and SM 5310C				
Alkalinity as CaCO ₃	3812-32-6	SM 2320B	5000	10000	7045	23483	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	250	500	---	---	---	---	---	---
Biochemical Oxygen Demand (BOD)	---	SM 5210B	2000	2000	---	---	---	---	---	---
Chloride	16887-00-6	SM 4500-Cl E	2000	4000	---	---	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	13450	44830	---	---	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	200	400	---	---	---	---	---	---
Hardness	---	SW846 6010	0.15	2.00	---	---	---	---	---	---
Methane	74-82.8	RSK-175	1.369684	2.80	---	---	---	---	---	---
Nitrate	14797-55-8	EPA Method 353.2	2000	4000	---	---	---	---	---	---
Nitrogen, Nitrate & Nitrite	14797-55-8 &14797-65-0	EPA Method 353.2	95	250	---	---	---	---	---	---
Oil & Grease	---	EPA Method 1664A	1130	5000	---	---	---	---	---	---
pH	---	EPA Method 150.1/ SM 9040/ SM 9045	0.01 SU	0.1 SU	---	---	---	---	---	---
Phosphate	14265-44-2	EPA Method 365.1	---	---	---	---	---	---	---	---
Residue, Non-filterable Total Suspended Solids (TSS)	---	SM 2540D	4750	10000	---	---	---	---	---	---
Sulfate	14808-79-8	SM 4500 SO4 E	2000	4000	---	---	---	---	---	---
Sulfur	7704-34-9	SW846 6010	36.1	500	---	---	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151	0.054	0.20	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060	254	850	252	840	---	---	---	---
Alternate Methods Provided										
Phenolics	Multiple	EPA Method 420.4	3.38	10.00	---	---	---	---	---	---
1,2-Dichloroethene	540-59-0	SW846-8260B	---	---	---	---	---	---	0.51	2
cis-1,2-Dichloroethene	156-59-2	SW846-8260B	---	---	---	---	---	---	0.25	1
trans-1,2-Dichloroethene	156-60-5	SW846-8260B	---	---	---	---	---	---	0.26	1
p-Isopropyltoluene	99-87-6	SW846-8260B	---	---	---	---	---	---	0.21	1
Dichloromethane	75-09-2	SW846-8260B	---	---	---	---	---	---	0.24	1
Chrome VI	18540-29-9	SM 3500 Cr-D	5.1	20	---	---	---	---	---	---
Mercury	7439-97-6	SW846 7470A	0.179	0.597	---	---	---	---	---	---
Chloride	16887-00-6	EPA Method 300.0 / SW846-9056A	2000	4000	---	---	---	---	---	---
Fluoride	16984-48-8	EPA Method 300.0 / SW846-9056A	200	400	---	---	---	---	---	---

Table 1D

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Methane	74-82-8	SW846-8015B	1.369684	2.80	---	---	---	---	---	---
Nitrate	14797-55-8	EPA Method 300.0 / SW846-9056A	2000	4000	---	---	---	---	---	---
Sulfate	14808-79-8	EPA Method 300.0 / SW846-9056A	2000	4000	---	---	---	---	---	---

O: SLM C: SSW F: SLM

Notes:
aka = also known as
CAS = Chemical Abstracts Service
CaCO₃ = Calcium Carbonate
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136
MDL = Method Detection Limit
MGP = Manufactured Gas Plant
OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
PAH = polycyclic aromatic hydrocarbon
PCBs = polychlorinated biphenyls
RAF = Multi-Site Risk Assessment Framework
RCRA = Resource Conservation and Recovery Act
RL = Reporting Limit
RSK-175: RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP = Standard operating procedure
SU= Standard Unit (pH is dimensionless)
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
TPH = Total Petroleum Hydrocarbons
USEPA or EPA = United States Environmental Protection Agency
VOA = volatile organic analyte/analysis
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
µg/L = micrograms per liter
--- = Lab does not offer analysis, analysis was not requested, no data
Bold Underline Text = Lab will provide an alternate method than what is identified in method column

^A Prior to any project-specific field data collection, the selected lab's ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.
^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for surface water and groundwater. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.
^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 2A. Multi-Site Program Soil/Sediment Matrix - Lowest Achievable Limits - Alpha, Brighton, STAT, Battelle, ESS, and EERC
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Alpha - Lowest Achievable Limits		Brighton - Lowest Achievable Limits		STAT - Lowest Achievable Limits		Battelle - Lowest Achievable Limits		ESS - Lowest Achievable Limits		EERC - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Petroleum Volatile Organic Compounds														
Benzene	71-43-2	SW846-8260B/ 8021B	9.65	50	18	50	0.2	5	---	---	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B/ 8021B	8.5	50	11	50	0.1	5	---	---	---	---	---	---
Toluene	108-88-3	SW846-8260B/ 8021B	9.75	75	20	50	0.2	5	---	---	---	---	---	---
Xylenes (Total)	1330-20-7	SW846-8260B/ 8021B	16.9	100	22	150	0.4	15	---	---	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B/ 8021B	8.05	250	10	50	0.2	5	---	---	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B/ 8021B	9.3	250	13	50	0.2	5	---	---	---	---	---	---
Diesel Range Organics (Total Petroleum Hydrocarbons (TPH)	---	WI DRO/8015C/8015D	1840	33400	400	4000.0	---	---	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	48.2	2500	400	4000.0	---	---	---	---	---	---	---	---
Semivolatile Organic Compounds														
Naphthalene	91-20-3	SW846-8270C/D/SIM PAH	0.289	1	5	330	24	33	0.210	1.25	0.342	4.0	0.11	0.02
C1-naphthalenes	---	SW846-8270C/D/SIM PAH	0.289	1	---	---	---	---	0.210	1.25	0.342	4.0	0.13	0.01
C2-naphthalenes	---	SW846-8270C/D/SIM PAH	0.289	1	---	---	---	---	0.210	1.25	0.342	4.0	0.15	0.03
C3-napthalenes	---	SW846-8270C/D/SIM PAH	0.289	1	---	---	---	---	0.210	1.25	0.342	4.0	0.17	0.04
C4-napthalenes	---	SW846-8270C/D/SIM PAH	0.289	1	---	---	---	---	0.210	1.25	0.342	4.0	0.19	0.04
Acenaphthylene	208-96-8	SW846-8270C/D/SIM PAH	0.192	1	9	330	22	33	0.378	1.25	0.530	4.0	0.13	0.01
Acenaphthene	83-32-9	SW846-8270C/D/SIM PAH	0.177	1	13	330	26	33	0.180	1.25	0.612	4.0	0.14	0.02
Fluorene	86-73-7	SW846-8270C/D/SIM PAH	0.268	1	15	330	29	33	0.204	1.25	0.375	4.0	0.16	0.02
C1-fluorenes	---	SW846-8270C/D/SIM PAH	0.268	1	---	---	---	---	0.204	1.25	0.375	4.0	0.18	0.04
C2-fluorenes	---	SW846-8270C/D/SIM PAH	0.268	1	---	---	---	---	0.204	1.25	0.375	4.0	0.20	0.04
C3-fluorenes	---	SW846-8270C/D/SIM PAH	0.268	1	---	---	---	---	0.204	1.25	0.375	4.0	0.23	0.04
Phenanthrene	85-01-8	SW846-8270C/D/SIM PAH	0.333	1	10	330	22	33	0.267	1.25	1.766	4.0	0.18	0.04
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.207	1	16	330	17	33	0.264	1.25	0.305	4.0	0.17	0.02
C1-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.333	1	---	---	---	---	0.267	1.25	1.766	4.0	0.20	0.04
C2-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.333	1	---	---	---	---	0.267	1.25	1.766	4.0	0.22	0.10
C3-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.333	1	---	---	---	---	0.267	1.25	1.766	4.0	0.24	0.05
C4-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.333	1	---	---	---	---	0.267	1.25	1.766	4.0	0.27	0.05
Fluoranthene	206-44-0	SW846-8270C/D/SIM PAH	0.319	1	10	330	20	33	0.426	1.25	0.688	4.0	0.21	0.04
Pyrene	129-00-0	SW846-8270C/D/SIM PAH	0.264	1	35	330	19	33	0.597	1.25	0.547	4.0	0.21	0.03
C1-pyrene/fluoranthenes	---	SW846-8270C/D/SIM PAH	0.264	1	---	---	---	---	0.597	1.25	0.547	4.0	0.23	0.02
Benzo(a)anthracene	56-55-3	SW846-8270C/D/SIM PAH	0.205	1	17	330	7	33	0.366	1.25	0.311	4.0	0.25	0.01
Chrysene	218-01-9	SW846-8270C/D/SIM PAH	0.203	1	16	330	19	33	0.633	1.25	0.498	4.0	0.25	0.01
C1-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.203	1	---	---	---	---	0.633	1.25	0.498	4.0	0.27	0.10
C2-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.203	1	---	---	---	---	0.633	1.25	0.498	4.0	0.30	0.10
C3-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.203	1	---	---	---	---	0.633	1.25	0.498	4.0	0.33	0.20
C4-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.203	1	---	---	---	---	0.633	1.25	0.498	4.0	0.36	0.20
Benzo(b)fluoranthene	205-99-2	SW846-8270C/D/SIM PAH	0.261	1	30	330	15	33	0.471	1.25	0.972	4.0	0.29	0.02
Benzo(k)fluoranthene	207-08-9	SW846-8270C/D/SIM PAH	0.199	1	25	330	16	33	0.639	1.25	0.706	4.0	0.29	0.02
Benzo(a)pyrene	50-32-8	SW846-8270C/D/SIM PAH	0.287	1	16	330	13	33	1.000	1.25	0.536	4.0	0.28	0.01
Perylene	198-55-0	SW846-8270C/D/SIM PAH	0.194	1	---	---	---	---	0.297	1.25	0.437	4.0	0.28	0.01
Benzo(e)pyrene	192-97-2	SW846-8270C/D/SIM PAH	0.207	1	---	---	---	---	0.495	1.25	0.721	4.0	0.28	0.01
Indeno(1,2,3-cd)pyrene	193-39-5	SW846-8270C/D/SIM PAH	0.273	1	24	330	12	33	0.507	1.25	2.585	4.0	0.33	0.01
Dibenzo(a,h)anthracene	53-70-3	SW846-8270C/D/SIM PAH	0.271	1	33	330	13	33	0.474	1.25	0.553	4.0	0.33	0.01
Benzo(g,h,i)perylene	191-24-2	SW846-8270C/D/SIM PAH	0.267	1	16	330	14	33	0.228	1.25	0.580	4.0	0.32	0.01
1-Methylnaphthalene	90-12-0	SW846-8270C/D/SIM PAH	0.317	1	---	---	7.2	170	0.228	1.25	0.391	4.0	0.13	0.01
2-Methylnaphthalene	91-57-6	SW846-8270C/D/SIM PAH	0.259	1	20	330	13	170	0.171	1.25	0.633	4.0	0.13	0.01
Carbazole	86-74-8	SW846-8270C/D/SIM PAH	0.329	1	29	330	19	170	---	---	58	333	---	---
Dibenzofuran	132-64-9	SW846-8270C/D/SIM PAH	0.316	1	27	330	12	170	0.174	1.25	0.441	4.0	---	---
Phenols														
2,4-dimethylphenol	105-67-9	SW846-8270C	5.49	33.3	18	330	26	170	---	---	75	333	---	---
2-methylphenol (o-cresol)	95-48-7	SW846-8270C	2.87	33.3	29	330	13	170	---	---	84	333	---	---
3&4-methylphenol (m, p-cresol)	106-44-5	SW846-8270C	4.81	33.3	29	330	40	170	---	---	177	667	---	---
phenol	108-95-2	SW846-8270C	3.17	33.3	4	330	50	170	---	---	81	333	---	---
Pesticides														
Chlordane	17289-03-6	SW846 8081A	20.1	20.1	1.0	25	3.1	16	---	---	---	---	---	---
Aldrin	309-00-2	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
α-BHC	319-84-6	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
β-BHC	319-85-7	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
δ-BHC	319-86-8	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081B	0.4	0.4	1.0	20	0.51	1.6	---	---	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Alpha - Lowest Achievable Limits		Brighton - Lowest Achievable Limits		STAT - Lowest Achievable Limits		Battelle - Lowest Achievable Limits		ESS - Lowest Achievable Limits		EERC - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
4,4'-DDE	72-55-9	SW846 8081B	0.4	0.4	1.0	20	0.6	1.6	---	---	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081B	0.4	0.4	1.0	20	0.56	1.6	---	---	---	---	---	---
Diallate	2303-16-4	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Dieldrin	60-57-1	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Endosulfan I	959-98-8	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Endrin	72-20-8	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Endrin ketone	53494-70-5	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Heptachlor	76-44-8	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Isodrin	465-73-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Methoxychlor	72-43-5	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
Toxaphene	8001-35-2	SW846 8081B	---	---	1.0	25	---	---	---	---	---	---	---	---
PCBs														
Aroclor 1016	12674-11-2	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
Aroclor 1260	11096-82-5	SW846 8082A	---	---	2.5	330	---	---	---	---	---	---	---	---
PCB Congeners														
2-Chlorobiphenyl	2051-60-7	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',5'-Trichlorobiphenyl	37680-65-2	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,4',5'-Trichlorobiphenyl	16606-02-3	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,3,3',4,6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
Indicator Parameters														
Soot Carbon	---	Lloyd Kahn Method	0.01%	0.01%	---	---	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060A	0.01%	0.01%	19	10000	---	---	---	---	---	---	---	---
Fraction Organic Carbon (FOC)	---	ASTM D2974	0.10%	0.10%	---	---	---	0.01%	---	---	---	---	---	---
Inorganics														
Aluminum	7429-90-5	SW846-6020A	1480	10000	32	1000	83	2000	---	---	---	---	---	---
Antimony	7440-36-0	SW846-6020A	13.52	160	16	500	42	200	---	---	---	---	---	---
Arsenic	7440-38-2	SW846-6020A	6.6	50	11	100	25	100	---	---	---	---	---	---
Barium	7440-39-3	SW846-6020A	21.1	300	7	1000	24	100	---	---	---	---	---	---
Cadmium	7440-43-9	SW846-6020A	2.64	20	10	50	11	50	---	---	---	---	---	---
Chromium (total)	7440-47-3	SW846-6020A	46.8	200	6	500	16	100	---	---	---	---	---	---
Copper	7440-50-8	SW846-6020A	19.4	200	32	1000	33	250	---	---	---	---	---	---
Cyanide, total	57-12-5	SW846-9012A	212	1000	20	100	35	250	---	---	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	125	1000	20	40	---	---	---	---	---	---	---	---
Iron	7439-89-6	SW846-6020A	2060	20000	290	2000	1070	3000	---	---	---	---	---	---
Lead	7439-92-1	SW846-6020A	14.6	60	6	1000	8.2	50	---	---	---	---	---	---
Manganese	7439-96-5	SW846-6020A	44.4	200	6	1000	26	100	---	---	---	---	---	---
Mercury	7439-97-6	SW846-7471A/B	1.6	12.5	1	50	0.6	20	---	---	---	---	---	---
Nickel	7440-2-0	SW846-6020A	26.7	100	270	1000	21	100	---	---	---	---	---	---
Selenium	7782-49-2	SW846-6020A	75.6	200	20	200	56	100	---	---	---	---	---	---
Silver	7440-22-4	SW846-6020A	4.88	50	2	500	3.4	100	---	---	---	---	---	---
Other														
Black carbon (Gustafsson)	---	Gustafsson Method	0.01%	0.01%	---	---	---	---	---	---	---	---	---	---
Alkalinity	3812-32-6	SM 2320B	---	---	50000	400000	---	---	---	---	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	<u>2790</u>	<u>7500</u>	400	1000	2170	2500	---	---	---	---	---	---
Biological Oxygen Demand (BOD)	---	SM 5210B	---	---	---	2000	---	---	---	---	---	---	---	---
British Therman Unit (BTU)	---	ASTM D240	---	---	---	200	---	---	---	---	---	---	---	---

Table 2A

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Alpha - Lowest Achievable Limits		Brighton - Lowest Achievable Limits		STAT - Lowest Achievable Limits		Battelle - Lowest Achievable Limits		ESS - Lowest Achievable Limits		EERC - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Bulk Density	---	ASTM D2937	0.01 lbs/ft3	0.01 lbs/ft3	---	1	---	---	---	---	---	---	---	---
Vanadium	7440-62-2	SW846-6020A	37.9	100	4	1000	30	100	---	---	---	---	---	---
Zinc	7440-66-6	SW846-6020A	260	1000	260	1000	80	500	---	---	---	---	---	---
Chloride	16887-00-6	EPA Method 325.2	---	---	6	20000	---	---	---	---	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	200000	200000	10000	60000	---	---	---	---	---	---	---	---
Cyanide, Reactive	57-12-5	SW846 7.3.3.2	10.00	10.00	---	50000	618.2	1000	---	---	---	---	---	---
Flash Point	---	EPA Method 1010/1020	---	70 Deg F	---	60-200 Deg F	---	---	---	---	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	1.04	10.00	30	4000	---	5000	---	---	---	---	---	---
Free Liquids (Paint Filter)	---	SW846 9095B	---	---	---	pass/fail	---	---	---	---	---	---	---	---
Moisture Content	---	SW846 3550C	0.001%	0.001%	---	%	---	0.01	---	---	---	---	---	---
Nitrate/Nitrite	14797-55-8 & 14797-65-0	EPA Method 353.2	2850	10000	10	1000	1000	2000	---	---	---	---	---	---
Total Kjeldahl Nitrogen	7727-37-9	SM 4500-NH3 G	31500	150000	100	1000	---	---	---	---	---	---	---	---
pH	---	SW846 9045D	---	---	---	2-12 SU	---	---	---	---	---	---	---	---
Phenolics	Multiple	SW846 9066	---	---	50	500	96	250	---	---	---	---	---	---
Phosphate	14265-44-2	EPA Method 365.1	15.0	15.0	---	---	---	---	---	---	---	---	---	---
Total Phosphorus	7723-14-0	EPA Method 365.1	1670	5000	20	200	---	---	---	---	---	---	---	---
Specific Gravity	---	ASTM D5057	---	---	---	---	---	---	---	---	---	---	---	---
Sulfate	14808-79-8	SM 4500-SO4 E	13600	100000	2000	20000	5470	50000	---	---	---	---	---	---
Sulfur	7704-34-9	SW846 6010	---	---	5000	50000	---	---	---	---	---	---	---	---
Sulfur, Reactive	7704-34-10	SW846 7.3.4.2	10.00	10.00	---	50000	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	ASTM-2974-00	---	---	---	---	---	0.01%	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-1	Lloyd Kahn Method	0.05%	0.05%	---	---	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-2	Walkley Black Method	---	---	---	---	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-3	SW846-9060M	0.01%	0.01%	---	---	---	---	---	---	---	---	---	---
Oil and Grease	---	SW846-9071B	200000	200000	---	---	129000	167000	---	---	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151/8321A/8270C/D	---	---	---	---	1.7	10	---	---	---	---	---	---
Alternate Methods Provided														
Ammonia	7644-41-7	SM 4500NH3	2790	7500	---	---	---	---	---	---	---	---	---	---
Moisture Content	---	SM 2540G	0.001%	0.001%	---	---	---	---	---	---	---	---	---	---
Nitrate/Nitrite	14797-55-8 & 14797-65-0	SM 4500NO3-F	2850	10000	---	---	---	---	---	---	---	---	---	---
Phosphate	14265-44-2	SM 4500P-E(M)	15	15	---	---	---	---	---	---	---	---	---	---
Total Phosphorus	7723-14-0	SM 4500P-E	1670	5000	---	---	---	---	---	---	---	---	---	---

Notes:
ASTM = ASTM International
CAS = Chemical Abstracts Service
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
Deg F = degrees Fahrenheit
dry wt. = dry weight
lbs/ft³ = pounds per cubic foot
MDL = Method Detection Limit
MGP = Manufactured Gas Plant
OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyls
RAF = Multi-Site Risk Assessment Framework
RCRA = Resource Conservation and Recovery Act
RL = Reporting Limit
RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP = Standard operating procedure
SU= Standard Unit (pH is dimensionless)
SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition
TPH= Total Petroleum Hydrocarbons
USEPA or EPA = United States Environmental Protection Agency
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
µg/kg = micrograms per kilogram
--- = Lab does not offer analysis, analysis was not requested, no data

Bold Underline Text = Lab will provide an alternate method than what is identified in method column

^A Prior to any project-specific field data collection, the selected lab's ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for soil and sediment. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

O: SLM C: SSW F: SLM

Table 2B. Multi-Site Program Soil/Sediment Matrix - Lowest Achievable Limits - Test America Only
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List B	CAS Number	Analytical Method Name/Number ^C	Test America - Lowest Achievable Limits Chicago		Test America - Lowest Achievable Limits Pittsburgh		Test America - Lowest Achievable Limits Nashville		Test America - Lowest Achievable Limits Knoxville		Test America - Lowest Achievable Limits Canton		Test America - Lowest Achievable Limits Burlington	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)
			μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)	μg/kg (dry wt.)
Petroleum Volatile Organic Compounds														
Benzene	71-43-2	SW846-8260B	0.51	2	---	---	---	---	---	---	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B	0.505	2	---	---	---	---	---	---	---	---	---	---
Toluene	108-88-3	SW846-8260B	0.957	2	---	---	---	---	---	---	---	---	---	---
Xylenes (Total)	1330-20-7	SW846-8260B	0.64	4	---	---	---	---	---	---	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B	0.752	2	---	---	---	---	---	---	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B	0.704	2	---	---	---	---	---	---	---	---	---	---
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	1600	4000	---	---	---	---	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	500	1500	---	---	---	---	---	---	---	---	---	---
Semivolatile Organic Compounds			Routine PAHs/SVOCs SW846-8270D		LL PAHs/SVOCs SW846-8270D		Alkylated/Forensic PAHs SW846-8270D SIM		LL Alkylated PAHs GC/MS Isotope Dilution					
Naphthalene	91-20-3	Refer to Column Header	5.11	33.0	2.40	6.70	3.3	3.3	5.30	20.0	---	---	---	---
C1-naphthalenes	---	Refer to Column Header	---	---	---	---	**	**	**	**	---	---	---	---
C2-naphthalenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	2.00	2.00	---	---	---	---
C3-naphthalenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	2.00	2.00	---	---	---	---
C4-naphthalenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
Acenaphthylene	208-96-8	Refer to Column Header	4.38	33.0	2.26	6.70	3.3	3.3	0.0630	1.00	---	---	---	---
Acenaphthene	83-32-9	Refer to Column Header	5.97	33.0	2.24	6.70	3.3	3.3	0.210	1.00	---	---	---	---
Fluorene	86-73-7	Refer to Column Header	4.67	33.0	2.38	6.70	5.0	5.0	0.470	1.00	---	---	---	---
C1-fluorenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C2-fluorenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C3-fluorenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
Phenanthrene	85-01-8	Refer to Column Header	4.63	33.0	2.79	6.70	3.3	3.3	1.60	2.00	---	---	---	---
Anthracene	120-12-7	Refer to Column Header	5.55	33.0	2.46	6.70	3.3	3.3	0.190	1.00	---	---	---	---
C1-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C2-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C3-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C4-phenanthrene/anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
Fluoranthene	206-44-0	Refer to Column Header	6.16	33.0	2.63	6.70	3.3	3.3	0.360	1.00	---	---	---	---
Pyrene	129-00-0	Refer to Column Header	6.60	33.0	2.97	6.70	3.3	3.3	1.10	2.00	---	---	---	---
C1-pyrene/fluoranthenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
Benzo(a)anthracene	56-55-3	Refer to Column Header	4.47	33.0	2.65	6.70	3.3	3.3	0.290	1.00	---	---	---	---
Chrysene	218-01-9	Refer to Column Header	9.06	33.0	2.60	6.70	3.3	3.3	0.200	1.00	---	---	---	---
C1-Chrysenes/Benz(a)anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C2-Chrysenes/Benz(a)anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C3-Chrysenes/Benz(a)anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
C4-Chrysenes/Benz(a)anthracenes	---	Refer to Column Header	---	---	---	---	3.3	3.3	1.00	1.00	---	---	---	---
Benzo(b)fluoranthene	205-99-2	Refer to Column Header	7.17	33.0	2.89	6.70	3.3	3.3	0.250	1.00	---	---	---	---
Benzo(k)fluoranthene	207-08-9	Refer to Column Header	9.79	33.0	2.31	6.70	3.3	3.3	0.220	1.00	---	---	---	---
Benzo(a)pyrene	50-32-8	Refer to Column Header	6.43	33.0	2.32	6.70	3.3	3.3	0.190	1.00	---	---	---	---
Perylene	198-55-0	Refer to Column Header	---	---	---	---	3.3	3.3	0.120	1.00	---	---	---	---
Benzo(e)pyrene	192-97-2	Refer to Column Header	---	---	2.60	33.0	3.3	3.3	0.170	1.00	---	---	---	---
Indeno(1,2,3-cd)pyrene	193-39-5	Refer to Column Header	8.61	33.0	2.58	6.70	3.3	3.3	0.170	1.00	---	---	---	---
Dibenzo(a,h)anthracene	53-70-3	Refer to Column Header	6.42	33.0	2.76	6.70	3.3	3.3	0.0700	1.00	---	---	---	---
Benzo(g,h,i)perylene	191-24-2	Refer to Column Header	10.7	33.0	2.89	6.70	3.3	3.3	0.150	1.00	---	---	---	---
1-Methylnaphthalene	90-12-0	Refer to Column Header	8.11	67.0	2.21	6.70	3.3	3.3	1.30	5.00	---	---	---	---
2-Methylnaphthalene	91-57-6	Refer to Column Header	6.11	67.0	2.56	6.70	3.3	3.3	2.90	10.0	---	---	---	---
Carbazole	86-74-8	Refer to Column Header	83.0	167	2.85	6.70	---	---	---	---	---	---	---	---
Dibenzofuran	132-64-9	Refer to Column Header	38.9	167	2.68	33.0	3.3	3.3	---	---	---	---	---	---
Phenols														
2,4-dimethylphenol	105-67-9	SW846-8270C	126	330	2.57	33.0	---	---	---	---	---	---	---	---
2-methylphenol (o-cresol)	95-48-7	SW846-8270C	53.3	167	2.94	33.0	---	---	---	---	---	---	---	---
3&4-methylphenol (m, p-cresol)	106-44-5	SW846-8270C	55.4	167	2.95	33.0	---	---	---	---	---	---	---	---
phenol	108-95-2	SW846-8270C	73.8	167	4.51	33.0	---	---	---	---	---	---	---	---
Pesticides														
Chlordane	57-74-9	SW846-8081A	3.25	6.7	---	---	---	---	---	---	---	---	---	---
Aldrin	309-00-2	SW846 8081B	0.693	1.7	---	---	---	---	---	---	---	---	---	---
α-BHC	319-84-6	SW846 8081B	0.424	1.7	---	---	---	---	---	---	---	---	---	---
β-BHC	319-85-7	SW846 8081B	0.518	1.7	---	---	---	---	---	---	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	0.362	1.7	---	---	---	---	---	---	---	---	---	---
δ-BHC	319-86-8	SW846 8081B	0.526	1.7	---	---	---	---	---	---	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081B	0.845	1.7	---	---	---	---	---	---	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081B	0.438	1.7	---	---	---	---	---	---	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	3.25	6.7	---	---	---	---	---	---	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---

Project Compound List B	CAS Number	Analytical Method Name/Number ^c	Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits		Test America - Lowest Achievable Limits	
			Chicago		Pittsburgh		Nashville		Knoxville		Canton		Burlington	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081B	0.333	1.7	---	---	---	---	---	---	---	---	---	---
4,4'-DDE	72-55-9	SW846 8081B	0.277	1.7	---	---	---	---	---	---	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081B	0.88	1.7	---	---	---	---	---	---	---	---	---	---
Diallate	2303-16-4	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Dieldrin	60-57-1	SW846 8081B	0.229	1.7	---	---	---	---	---	---	---	---	---	---
Endosulfan I	959-98-8	SW846 8081B	0.731	1.7	---	---	---	---	---	---	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081B	0.271	1.7	---	---	---	---	---	---	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	0.305	1.7	---	---	---	---	---	---	---	---	---	---
Endrin	72-20-8	SW846 8081B	0.231	1.7	---	---	---	---	---	---	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	0.281	1.7	---	---	---	---	---	---	---	---	---	---
Endrin ketone	53494-70-5	SW846 8081B	0.378	1.7	---	---	---	---	---	---	---	---	---	---
Heptachlor	76-44-8	SW846 8081B	0.701	1.7	---	---	---	---	---	---	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	0.594	1.7	---	---	---	---	---	---	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Isodrin	465-73-6	SW846 8081B	---	---	---	---	---	---	---	---	---	---	---	---
Methoxychlor	72-43-5	SW846 8081B	0.324	8.3	---	---	---	---	---	---	---	---	---	---
Toxaphene	8001-35-2	SW846 8081B	7.04	16.7	---	---	---	---	---	---	---	---	---	---
PCBs														
Aroclor 1016	12674-11-2	SW846 8082A	5.9	16.7	---	---	---	---	---	---	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	7.34	16.7	---	---	---	---	---	---	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	7.27	16.7	---	---	---	---	---	---	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	5.48	16.7	---	---	---	---	---	---	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	6.57	16.7	---	---	---	---	---	---	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	3.6	16.7	---	---	---	---	---	---	---	---	---	---
Aroclor 1260	11096-82-5	SW846 8082A	8.19	16.7	---	---	---	---	---	---	---	---	---	---
PCB Congeners														
2-Chlorobiphenyl	2051-60-7	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	0.616	1	---	---	---	---	---	---	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	0.393	1	---	---	---	---	---	---	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	0.401	1	---	---	---	---	---	---	---	---	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	0.349	1	---	---	---	---	---	---	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	0.251	1	---	---	---	---	---	---	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	0.274	1	---	---	---	---	---	---	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	0.182	1	---	---	---	---	---	---	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	---	---	---	---	---	---	---	---	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	0.235	1	---	---	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	0.208	1	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	0.106	1	---	---	---	---	---	---	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	0.657	1	---	---	---	---	---	---	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	0.255	1	---	---	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	0.453	1	---	---	---	---	---	---	---	---	---	---
Indicator Parameters														
Soot Carbon	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846-9060A/ASTM D4129-82M/ASTM D2974/Lloyd Kahn Method	---	---	---	---	---	---	---	---	---	---	380000	1000000
Fractional Organic Carbon (FOC)	---	SW846-9060A/ASTM D4129-82M/ASTM D2974/Lloyd Kahn Method	0.058 wt%	0.058 wt%	---	---	---	---	---	---	---	---	---	---
Inorganics														
			Inorganics SW846-6010B		Inorganics SW846-6020A									
Aluminum	7429-90-5	Refer to Column Header	8170	20000	1820	3000	---	---	---	---	---	---	---	---
Antimony	7440-36-0	Refer to Column Header	389	2000	32.5	200	---	---	---	---	---	---	---	---
Arsenic	7440-38-2	Refer to Column Header	342	1000	20.3	100	---	---	---	---	---	---	---	---
Barium	7440-39-3	Refer to Column Header	114	1000	47.4	1000	---	---	---	---	---	---	---	---
Cadmium	7440-43-9	Refer to Column Header	36	200	10.5	100	---	---	---	---	---	---	---	---
Chromium (total)	7440-47-3	Refer to Column Header	495	1000	81.6	200	---	---	---	---	---	---	---	---
Copper	7440-50-8	Refer to Column Header	280	1000	122	200	---	---	---	---	---	---	---	---
Cyanide, total	57-12-5	SW846-9012A/9014	172	500	153	500	---	---	---	---	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	---	---	22	40	---	---	---	---	---	---	---	---
Iron	7439-89-6	Refer to Column Header	10400	20000	3680	5000	---	---	---	---	---	---	---	---
Lead	7439-92-1	Refer to Column Header	231	500	48.1	100	---	---	---	---	---	---	---	---
Manganese	7439-96-5	Refer to Column Header	145	1000	174	500	---	---	---	---	---	---	---	---
Mercury	7439-97-6	SW846-7471A/B	5.56	16.7	7.39	33	---	---	---	---	---	---	---	---

Table 2B

Project Compound List B	CAS Number	Analytical Method Name/Number ^C	Test America - Lowest Achievable Limits	Test America - Lowest Achievable Limits	Test America - Lowest Achievable Limits	Test America - Lowest Achievable Limits	Test America - Lowest Achievable Limits	Test America - Lowest Achievable Limits						
			Chicago		Pittsburgh		Nashville		Knoxville		Canton		Burlington	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Nickel	7440-2-0	Refer to Column Header	291	1000	29.4	100	---	---	---	---	---	---	---	---
Selenium	7782-49-2	Refer to Column Header	588	1000	122	500	---	---	---	---	---	---	---	---
Silver	7440-22-4	Refer to Column Header	129	500	13.3	100	---	---	---	---	---	---	---	---
Vanadium	7440-62-2	Refer to Column Header	118	500	56.5	100	---	---	---	---	---	---	---	---
Zinc	7440-66-6	Refer to Column Header	878	2000	288	500	---	---	---	---	---	---	---	---
Other														
Black carbon (Gustafsson)	---	Gustafsson Method	---	---	---	---	---	---	---	---	---	---	1000000	1000000
Alkalinity	3812-32-6	SM 2320B	374000	500000	---	---	---	---	---	---	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	10000	20000	---	---	---	---	---	---	---	---	---	---
Biological Oxygen Demand (BOD)	---	SM 5210B	2000	2000	---	---	---	---	---	---	---	---	---	---
British Therman Unit (BTU)	---	ASTM D240	---	---	---	---	200	---	---	---	---	---	---	---
Bulk Density	---	SM2710F	0.1 g/cc	0.1 g/cc	---	---	---	---	---	---	---	---	---	---
Chloride	16887-00-6	SW846-9056A	1700	2000	---	---	---	---	---	---	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	24000	40000	---	---	---	---	---	---	---	---	---	---
Cyanide, Reactive (Total)	57-12-5	SW846-9010B/9014	172	500	---	---	---	---	---	---	---	---	---	---
Flash Point	---	EPA Method 1010/1020	40 Deg F	40 Deg F	---	---	---	---	---	---	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	563.00	1000.00	---	---	---	---	---	---	---	---	---	---
Free Liquids (Paint Filter)	---	SW846-9095B	0 mL/100g	0 mL/100g	---	---	---	---	---	---	---	---	---	---
Moisture Content	---	SW846-3550C	0.1%	0.1%	---	---	---	---	---	---	---	---	---	---
Nitrate/Nitrite	14797-55-8 & 14797-65-0	EPA Method 353.2	411	1000	---	---	---	---	---	---	---	---	---	---
Total Kjeldahl Nitrogen	7727-37-9	SM 4500-NH3 G	29000	40000	---	---	---	---	---	---	---	---	---	---
pH	---	SW846-9045D	0.2 SU	0.2 SU	---	---	---	---	---	---	---	---	---	---
Phenolics	Multiple	SW846-9066	411	500	---	---	---	---	---	---	---	---	---	---
Phosphate	14265-44-2	SM4500_P_E	6400	30600	---	---	---	---	---	---	---	---	---	---
Total Phosphorus	7723-14-0	SM4500_P_E	2090	10000	---	---	---	---	---	---	---	---	---	---
Specific Gravity	---	SM2710F	0.1	0.1	---	---	---	---	---	---	---	---	---	---
Sulfate	14808-79-8	SW846-9056A	950.0	2000.0	---	---	---	---	---	---	---	---	---	---
Sulfur	7704-34-9	SW846-6010	---	---	---	---	26000	50000	---	---	---	---	---	---
Sulfur, Reactive	7704-34-10	SW846 7.3.4.2	---	---	---	---	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-1	Lloyd Kahn Method	---	---	---	---	---	---	---	---	---	---	380000	1000000
Total Organic Carbon (TOC)	7440-44-2	Walkley Black Method	---	---	---	---	---	---	---	347000	1000000	---	---	---
Total Organic Carbon (TOC)	7440-44-3	SW846-9060M	---	---	---	---	600000	1000000	---	---	---	---	---	---
Oil and Grease	---	SW846-9071B	145000	240000	---	---	---	---	---	---	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151	21.70	33.0	---	---	---	---	---	---	---	---	---	---

Notes:

ASTM = ASTM International

CAS = Chemical Abstracts Service

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act

Deg F = degrees Fahrenheit

dry wt. = dry weight

g= grams

g/cc = grams per cubic centimeter

GC= gas chromatography

LL = low level

MDL = Method Detection Limit

MGP = Manufactured Gas Plants

mL= milliliter

MS = mass spectrometry

OIA 1667 = OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCBs = polychlorinated biphenyls

RAF = Multi-Site Risk Assessment Framework

RCRA = Resource Conservation and Recovery Act

RL = Reporting Limit

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP = Standard operating procedure

SU= Standard Unit (pH is dimensionless)

SVOC = semivolatile organic carbon

SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition

TPH= Total Petroleum Hydrocarbons

USEPA or EPA = United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

µg/kg = micrograms per kilogram

wt% = percentage by weight

--- = Lab does not offer analysis, analysis was not requested, no data

** Note from lab: C1-naphthalenes includes 1-methylnapthalene and 2-methylnapthalene, see additional table entries for these analytes.

^A Prior to any project-specific field data collection, the selected lab’s ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for soil and sediment. Other project-specific analytes of interest will be listed in Site-Specific Work

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 2C. Multi-Site Program Soil/Sediment Matrix - Lowest Achievable Limits - Eurofins Only
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Petroleum Volatile Organic Compounds						
Benzene	71-43-2	SW846-8260B/ 8021B	0.5	5	---	---
Ethylbenzene	100-41-4	SW846-8260B/ 8021B	1	5	---	---
Toluene	108-88-3	SW846-8260B/ 8021B	1	5	---	---
Xylenes (Total)	1330-20-7	SW846-8260B/ 8021B	1	5	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B/ 8021B	1	5	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B/ 8021B	1	5	---	---
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	---	---	---	---
Semivolatile Organic Compounds						
Naphthalene	91-20-3	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C1-naphthalenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C2-naphthalenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C3-naphthalenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C4-naphthalenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Acenaphthylene	208-96-8	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Acenaphthene	83-32-9	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Fluorene	86-73-7	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C1-fluorenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C2-fluorenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C3-fluorenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Phenanthrene	85-01-8	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C1-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C2-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C3-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C4-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Fluoranthene	206-44-0	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Pyrene	129-00-0	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C1-pyrene/fluoranthenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Benzo(a)anthracene	56-55-3	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Chrysene	218-01-9	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C1-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C2-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C3-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
C4-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Benzo(b)fluoranthene	205-99-2	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Benzo(k)fluoranthene	207-08-9	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Benzo(a)pyrene	50-32-8	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Perylene	198-55-0	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Benzo(e)pyrene	192-97-2	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Indeno(1,2,3-cd)pyrene	193-39-5	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Dibenzo(a,h)anthracene	53-70-3	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Benzo(g,h,i)perylene	191-24-2	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
1-Methylnaphthalene	90-12-0	SW846-8270C/D/SIM PAH	0.67	1.7	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
2-Methylnaphthalene	91-57-6	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Carbazole	86-74-8	SW846-8270C/D/SIM PAH	17	33	---	---
Dibenzofuran	132-64-9	SW846-8270C/D/SIM PAH	0.67	1.7	---	---
Phenols						
2,4-dimethylphenol	105-67-9	SW846-8270C	17	33	---	---
2-methylphenol (o-cresol)	95-48-7	SW846-8270C	17	33	---	---
3&4-methylphenol (m, p-cresol)	106-44-5	SW846-8270C	17	33	---	---
phenol	108-95-2	SW846-8270C	17	33	---	---
Pesticides						
Aldrin	309-00-2	SW846 8081B	0.17	0.83	---	---
α-BHC	319-84-6	SW846 8081B	0.17	0.83	---	---
β-BHC	319-85-7	SW846 8081B	0.3	1	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	0.17	0.83	---	---
δ-BHC	319-86-8	SW846 8081B	0.45	0.9	---	---
cis-Chlordane	5103-71-9	SW846 8081B	0.17	0.83	---	---
trans-Chlordane	5103-74-2	SW846 8081B	0.25	0.83	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	4	17	---	---
Chlorobenzilate	510-15-6	SW846 8081B	33	167	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	2	5	---	---
4,4'-DDD	72-54-8	SW846 8081B	0.33	1.7	---	---
4,4'-DDE	72-55-9	SW846 8081B	0.33	1.7	---	---
4,4'-DDT	50-29-3	SW846 8081B	0.36	1.7	---	---
Diallate	2303-16-4	SW846 8081B	33	167	---	---
Dieldrin	60-57-1	SW846 8081B	0.33	1.7	---	---
Endosulfan I	959-98-8	SW846 8081B	0.22	0.83	---	---
Endosulfan II	33213-65-9	SW846 8081B	0.33	1.7	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	0.33	1.7	---	---
Endrin	72-20-8	SW846 8081B	0.34	1.7	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	0.33	1.7	---	---
Endrin ketone	53494-70-5	SW846 8081B	0.6	1.8	---	---
Heptachlor	76-44-8	SW846 8081B	0.17	0.83	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	0.17	0.83	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	0.21	0.83	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	167	500	---	---
Isodrin	465-73-6	SW846 8081B	17	33	---	---
Methoxychlor	72-43-5	SW846 8081B	1.7	6.7	---	---
Toxaphene	8001-35-2	SW846 8081B	14	33	---	---
PCBs						
Aroclor 1016	12674-11-2	SW846 8082A	3.6	17	---	---
Aroclor 1221	11104-28-2	SW846 8082A	4.6	17	---	---
Aroclor 1232	11141-16-5	SW846 8082A	8	17	---	---
Aroclor 1242	53469-21-9	SW846 8082A	3.3	17	---	---
Aroclor 1248	12672-29-6	SW846 8082A	3.3	17	---	---
Aroclor 1254	11097-69-1	SW846 8082A	3.3	17	---	---
Aroclor 1260	11096-82-5	SW846 8082A	4.9	17	---	---
PCB Congeners						
2-Chlorobiphenyl	2051-60-7	SW846 8082A	1	2	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	0.8	5	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	1.6	5	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	1.8	5	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	4	10	---	---

Table 2C

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	<u>1.5</u>	<u>5</u>	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	<u>1.7</u>	<u>5</u>	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	<u>7.4</u>	<u>20</u>	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	<u>4.7</u>	<u>20</u>	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	<u>3.9</u>	<u>10</u>	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	<u>6.3</u>	<u>20</u>	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	<u>1.7</u>	<u>5</u>	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	<u>4.6</u>	<u>10</u>	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	<u>3</u>	<u>10</u>	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	<u>1.2</u>	<u>5</u>	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	<u>3</u>	<u>10</u>	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	<u>2.8</u>	<u>10</u>	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	<u>1.7</u>	<u>5</u>	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	<u>1.6</u>	<u>5</u>	---	---
Indicator Parameters						
Soot Carbon	---	Lloyd Kahn Method	3.3-8	17	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060A	100000	300000	---	---
Fraction Organic Carbon (FOC)	---	ASTM D2974	S	300000	---	---
Inorganics			SW846-6010B and 7471A	SW846-6010B and 7471A	SW846-6010C/D and 7471B	SW846-6010C/D and 7471B
Aluminum	7429-90-5	SW846-6020A	8940	20000	8940	40000
Antimony	7440-36-0	SW846-6020A	870	2000	870	4000
Arsenic	7440-38-2	SW846-6020A	960	2000	960	4000
Barium	7440-39-3	SW846-6020A	44	500	44	1000
Cadmium	7440-43-9	SW846-6020A	54	500	54	1000
Chromium (total)	7440-47-3	SW846-6020A	170	1500	170	3000
Copper	7440-50-8	SW846-6020A	240	1000	240	2000
Cyanide, total	57-12-5	SW846-9012A	500	180	---	---
Cyanide, available	57-12-5	OIA-1677	---	---	---	---
Iron	7439-89-6	SW846-6020A	8050	20000	8050	40000
Lead	7439-92-1	SW846-6020A	600	1500	600	3000
Manganese	7439-96-5	SW846-6020A	83	500	83	1000
Mercury	7439-97-6	SW846-7471A/B	10	100	10	100
Nickel	7440-2-0	SW846-6020A	150	1000	150	2000
Selenium	7782-49-2	SW846-6020A	930	2000	930	4000
Silver	7440-22-4	SW846-6020A	240	500	240	1000
Vanadium	7440-62-2	SW846-6020A	150	500	150	1000
Zinc	7440-66-6	SW846-6020A	240	2000	240	4000
Other						
Black carbon (Gustafsson)	---	Gustafsson Method	---	---	---	---
Alkalinity	3812-32-6	SM 2320B	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	2500	5000	---	---
Biological Oxygen Demand (BOD)	---	SM 5210B	---	---	---	---
British Therman Unit (BTU)	---	ASTM D240	---	---	---	---
Bulk Density	---	ASTM D2937	---	---	---	---
Chloride	16887-00-6	EPA Method 325.2	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	---	---	---	---
Cyanide, Reactive	57-12-5	SW846 7.3.3.2	20000	60000	---	---
Flash Point	---	EPA Method 1010/1020	50 Degrees F	50 Degrees F	---	---
Fluoride	16984-48-8	SM 4500F-C	30	100	---	---

Table 2C

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Eurofins - Lowest Achievable Limits		Eurofins - Lowest Achievable Limits	
			MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Free Liquids (Paint Filter)	---	SW846 9095B	Pos/Neg	Pos/Neg	---	---
Moisture Content	---	SW846 3550C	0.50%	0.50%	---	---
Nitrate/Nitrite	14797-55-8 &14797-65-0	EPA Method 353.2	---	---	---	---
Total Kjeldahl Nitrogen	7727-37-9	SM 4500-NH3 G	50000	160000	---	---
pH	---	SW846 9045D	0.01 SU	0.01 SU	---	---
Phenolics	Multiple	SW846 9066	1200	3500	---	---
Phosphate	14265-44-2	EPA Method 365.1	30700	61400	---	---
Total Phosphorus	7723-14-0	EPA Method 365.1	10000	20000	---	---
Specific Gravity	---	ASTM D5057	---	---	---	---
Sulfate	14808-79-8	SM 4500-SO4 E	---	---	---	---
Sulfur	7704-34-9	SW846 6010	8330	50000	---	---
Sulfur, Reactive	7704-34-10	SW846 7.3.4.2	53600	160000	---	---
Total Organic Carbon (TOC)	7440-44-0	ASTM-2974-00	---	---	---	---
Total Organic Carbon (TOC)	7440-44-1	Lloyd Kahn Method	100000	300000	---	---
Total Organic Carbon (TOC)	7440-44-2	Walkley Black Method	---	---	---	---
Total Organic Carbon (TOC)	7440-44-3	SW846 9060M	100000	300000	---	---
Oil and Grease	---	SW846-9071B	200000	600000	---	---
Pentachlorophenol	87-86-5	SW846-8151	0.3	1.7	---	---
Alternate Methods Provided						
Moisture Content	---	SM 2540G	0.50%	0.50%	---	---
Total Kjeldahl Nitrogen	7727-37-9	EPA Method 351.2	50000	160000	---	---
Chlorobenzilate	510-15-6	Method SW-846 8270	33	167	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	Method SW-846 8260	2	5	---	---
Diallate	2303-16-4	Method SW-846 8270	33	167	---	---
Hexachlorocyclopentadiene	77-47-4	Method SW-846 8270	167	500	---	---
Isodrin	465-73-6	Method SW-846 8270	17	33	---	---
2-Chlorobiphenyl	2051-60-7	EPA Method 1668A or C	1	2	---	---
2,3-Dichlorobiphenyl	16605-91-7	EPA Method 1668A or C	0.8	5	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	EPA Method 1668A or C	1.6	5	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	EPA Method 1668A or C	1.8	5	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	EPA Method 1668A or C	4	10	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	EPA Method 1668A or C	1.5	5	---	---
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	EPA Method 1668A or C	1.7	5	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	EPA Method 1668A or C	7.4	20	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	EPA Method 1668A or C	4.7	20	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	EPA Method 1668A or C	3.9	10	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	EPA Method 1668A or C	6.3	20	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	EPA Method 1668A or C	1.7	5	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	EPA Method 1668A or C	4.6	10	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	EPA Method 1668A or C	3	10	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	EPA Method 1668A or C	1.2	5	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	EPA Method 1668A or C	3	10	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	EPA Method 1668A or C	2.8	10	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	EPA Method 1668A or C	1.7	5	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	EPA Method 1668A or C	1.6	5	---	---

O: SLM

C: SSW

F: SLM

Notes:

ASTM = ASTM International

CAS = Chemical Abstracts Service

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act

Deg F = degrees Fahrenheit

dry wt. = dry weight
MDL = Method Detection Limit
MGP = Manufactured Gas Plant
OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl
RAF = Multi-Site Risk Assessment Framework
RCRA = Resource Conservation and Recovery Act
RL = Reporting Limit
RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP = Standard operating procedure
SU= Standard Unit (pH is dimensionless)
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
TPH= Total Petroleum Hydrocarbons
USEPA or EPA = United States Environmental Protection Agency
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
µg/kg = micrograms per kilogram
--- = Lab does not offer analysis, analysis was not requested, no data
Bold Underline Text = Lab will provide an alternate method than what is identified in method column

^A Prior to any project-specific field data collection, the selected lab’s ability to achieve approved project-specific screening levels as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for soil and sediment. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 2D. Multi-Site Program Soil/Sediment Matrix - Lowest Achievable Limits - Pace Analytical Only
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Petroleum Volatile Organic Compounds			8260	8260	8021	8021				
Benzene	71-43-2	SW846-8260B/ 8021B	25.00	50.0	10.00	20.0	---	---	---	---
Ethylbenzene	100-41-4	SW846-8260B/ 8021B	25.00	50.0	25.00	50.0	---	---	---	---
Toluene	108-88-3	SW846-8260B/ 8021B	25.00	50.0	25.00	50.0	---	---	---	---
Xylenes (Total)	1330-20-7	SW846-8260B/ 8021B	75.00	150.0	75.00	150.0	---	---	---	---
1,3,5-Trimethylbenzene	108-67-8	SW846-8260B/ 8021B	25.00	50.0	25.00	50.0	---	---	---	---
1,2,4-Trimethylbenzene	95-63-6	SW846-8260B/ 8021B	25.00	50.0	25.00	50.0	---	---	---	---
Diesel Range Organics (TPH)	---	WI DRO/8015C/8015D	1.30	4.4	---	---	---	---	---	---
Gasoline Range Organics (TPH)	---	WI DRO/8015C/8015D	1590.45	5000.0	---	---	---	---	---	---
Semivolatile Organic Compounds			8270 SIM	8270 SIM	8270	8270	Alkylated	Alkylated		
Naphthalene	91-20-3	SW846-8270C/D/SIM PAH	8.42	28.1	58.3639	194.55	0.660	60	0.25	2
C1-naphthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C2-naphthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C3-naphthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C4-naphthalenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
Acenaphthylene	208-96-8	SW846-8270C/D/SIM PAH	3.30	11.0	59.5	198.5	0.370	60	0.15	2
Acenaphthene	83-32-9	SW846-8270C/D/SIM PAH	3.88	12.9	59.1931	197.31	0.310	60	0.16	2
Fluorene	86-73-7	SW846-8270C/D/SIM PAH	4.14	13.8	15.5098	65.03	0.379	60	0.17	2
C1-fluorenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C2-fluorenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C3-fluorenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
Phenanthrene	85-01-8	SW846-8270C/D/SIM PAH	11.6	38.8	21.4147	71.38	0.930	60	0.25	2
Anthracene	120-12-7	SW846-8270C/D/SIM PAH	5.71	19.0	26.7	88.9	0.440	60	0.22	2
C1-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C2-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C3-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C4-phenanthrene/anthracenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
Fluoranthene	206-44-0	SW846-8270C/D/SIM PAH	5.21	17.4	23.6189	78.73	0.530	60	0.30	2
Pyrene	129-00-0	SW846-8270C/D/SIM PAH	4.51	15.0	37.0	123.3	0.460	60	0.27	2
C1-pyrene/fluoranthenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
Benzo(a)anthracene	56-55-3	SW846-8270C/D/SIM PAH	3.17	10.6	25.8504	86.168	0.342	60	0.35	2
Chrysene	218-01-9	SW846-8270C/D/SIM PAH	3.37	11.2	24.9569	83.19	0.339	60	0.16	3
C1-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C2-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C3-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
C4-benzo(a)anthracene/chrysenes	---	SW846-8270C/D/SIM PAH	---	---	---	---	5.000	60	---	---
Benzo(b)fluoranthene	205-99-2	SW846-8270C/D/SIM PAH	2.82	9.41	28.68	95.6	0.410	60	0.35	2.5
Benzo(k)fluoranthene	207-08-9	SW846-8270C/D/SIM PAH	2.51	8.36	39.9665	133.22	0.538	60	0.24	2.5
Benzo(a)pyrene	50-32-8	SW846-8270C/D/SIM PAH	2.51	8.37	25.1155	83.718	0.341	60	0.48	2
Perylene	198-55-0	SW846-8270C/D/SIM PAH	---	---	---	---	0.460	60	---	---
Benzo(e)pyrene	192-97-2	SW846-8270C/D/SIM PAH	2.66	8.87	---	---	0.441	60	---	---
Indeno(1,2,3-cd)pyrene	193-39-5	SW846-8270C/D/SIM PAH	2.20	7.33	36.1176	120.39	0.270	60	0.63	2
Dibenzo(a,h)anthracene	53-70-3	SW846-8270C/D/SIM PAH	2.24	7.45	45.3374	151.12	0.380	60	---	---
Benzo(g,h,i)perylene	191-24-2	SW846-8270C/D/SIM PAH	2.03	6.77	43.6689	145.56	0.330	60	---	---
1-Methylnaphthalene	90-12-0	SW846-8270C/D/SIM PAH	4.02	13.4	47.5	158.4	0.378	60	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
2-Methylnaphthalene	91-57-6	SW846-8270C/D/SIM PAH	5.00	16.7	43.3	144.5	0.408	60	---	---
Carbazole	86-74-8	SW846-8270C/D/SIM PAH	---	---	26.1	87.1	---	---	0.4	17
Dibenzofuran	132-64-9	SW846-8270C/D/SIM PAH	---	---	20.2	67.4	---	---	0.49	17
Phenols										
2,4-dimethylphenol	105-67-9	SW846-8270C	33.0074	110.02	---	---	---	---	3.6	170
2-methylphenol (o-cresol)	95-48-7	SW846-8270C	30.3264	101.09	---	---	---	---	3.6	17
3&4-methylphenol (m, p-cresol)	106-44-5	SW846-8270C	30.5892	101.96	---	---	---	---	3.7	17
phenol	108-95-2	SW846-8270C	39.612	132.04	---	---	---	---	3.9	170
Pesticides										
Aldrin	309-00-2	SW846 8081B	0.546	1.82	---	---	---	---	---	---
α-BHC	319-84-6	SW846 8081B	0.466	1.55	---	---	---	---	---	---
β-BHC	319-85-7	SW846 8081B	0.54	1.8	---	---	---	---	---	---
γ-BHC (Lindane)	58-89-9	SW846 8081B	1.054	3.51	---	---	---	---	---	---
δ-BHC	319-86-8	SW846 8081B	0.508	1.69	---	---	---	---	---	---
cis-Chlordane	5103-71-9	SW846 8081B	0.555	1.85	---	---	---	---	---	---
trans-Chlordane	5103-74-2	SW846 8081B	0.639	2.13	---	---	---	---	---	---
Chlordane -- not otherwise specified (n.o.s.)	57-74-9	SW846 8081B	9.814	32.7	---	---	---	---	---	---
Chlorobenzilate	510-15-6	SW846 8081B	---	---	---	---	---	---	---	---
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	SW846 8081B	---	---	---	---	---	---	---	---
4,4'-DDD	72-54-8	SW846 8081B	1.02	3.4	---	---	---	---	---	---
4,4'-DDE	72-55-9	SW846 8081B	1.085	3.62	---	---	---	---	---	---
4,4'-DDT	50-29-3	SW846 8081B	1.6	5.33	---	---	---	---	---	---
Diallate	2303-16-4	SW846 8081B	---	---	---	---	---	---	---	---
Dieldrin	60-57-1	SW846 8081B	1.047	3.49	---	---	---	---	---	---
Endosulfan I	959-98-8	SW846 8081B	0.525	1.75	---	---	---	---	---	---
Endosulfan II	33213-65-9	SW846 8081B	1.328	4.43	---	---	---	---	---	---
Endosulfan sulfate	1031-07-8	SW846 8081B	1.284	4.28	---	---	---	---	---	---
Endrin	72-20-8	SW846 8081B	1.177	3.92	---	---	---	---	---	---
Endrin aldehyde	7421-93-4	SW846 8081B	1.173	3.91	---	---	---	---	---	---
Endrin ketone	53494-70-5	SW846 8081B	1.745	5.82	---	---	---	---	---	---
Heptachlor	76-44-8	SW846 8081B	0.584	1.95	---	---	---	---	---	---
Heptachlor epoxide	1024-57-3	SW846 8081B	0.493	1.64	---	---	---	---	---	---
Hexachlorobenzene	118-74-1	SW846 8081B	0.676	2.25	---	---	---	---	---	---
Hexachlorocyclopentadiene	77-47-4	SW846 8081B	---	---	---	---	---	---	---	---
Isodrin	465-73-6	SW846 8081B	---	---	---	---	---	---	---	---
Methoxychlor	72-43-5	SW846 8081B	7.521	25.1	---	---	---	---	---	---
Toxaphene	8001-35-2	SW846 8081B	14.000	100.00	---	---	---	---	---	---
PCBs										
Aroclor 1016	12674-11-2	SW846 8082A	25	50	---	---	---	---	---	---
Aroclor 1221	11104-28-2	SW846 8082A	25	50	---	---	---	---	---	---
Aroclor 1232	11141-16-5	SW846 8082A	25	50	---	---	---	---	---	---
Aroclor 1242	53469-21-9	SW846 8082A	25	50	---	---	---	---	---	---
Aroclor 1248	12672-29-6	SW846 8082A	25	50	---	---	---	---	---	---
Aroclor 1254	11097-69-1	SW846 8082A	25	50	---	---	---	---	---	---
Aroclor 1260	11096-82-5	SW846 8082A	25	50	---	---	---	---	---	---
PCB Congeners										
2-Chlorobiphenyl	2051-60-7	SW846 8082A	---	---	---	---	---	---	---	---
2,3-Dichlorobiphenyl	16605-91-7	SW846 8082A	---	---	---	---	---	---	---	---
2,2',5-Trichlorobiphenyl	37680-65-2	SW846 8082A	---	---	---	---	---	---	---	---
2,4',5-Trichlorobiphenyl	16606-02-3	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,5'-Tetrachlorobiphenyl	41464-39-5	SW846 8082A	---	---	---	---	---	---	---	---
2,2',5,5'-Tetrachlorobiphenyl	35693-99-3	SW846 8082A	---	---	---	---	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
2,3',4,4'-Tetrachlorobiphenyl	32598-10-0	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,5'-Pentachlorobiphenyl	38380-02-8	SW846 8082A	---	---	---	---	---	---	---	---
2,2',4,5,5'-Pentachlorobiphenyl	37680-73-2	SW846 8082A	---	---	---	---	---	---	---	---
2,3,3',4',6-Pentachlorobiphenyl	38380-03-9	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,4',5'-Hexachlorobiphenyl	35065-28-2	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,5,5'-Hexachlorobiphenyl	52712-04-6	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,5,5',6-Hexachlorobiphenyl	52663-63-5	SW846 8082A	---	---	---	---	---	---	---	---
2,2',4,4',5,5'-Hexachlorobiphenyl	35065-27-1	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5-Heptachlorobiphenyl	35065-30-6	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,4',5,5'-Heptachlorobiphenyl	35065-29-3	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4,4',5',6-Heptachlorobiphenyl	52663-69-1	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,4',5,5',6-Heptachlorobiphenyl	52663-68-0	SW846 8082A	---	---	---	---	---	---	---	---
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	40186-72-9	SW846 8082A	---	---	---	---	---	---	---	---
Indicator Parameters										
Soot Carbon	---	Lloyd Kahn Method	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	SW846 9060A	193970	646600	---	---	---	---	---	---
Fraction Organic Carbon (FOC)	---	ASTM D2974	0.058 %w/w	0.058 %w/w	---	---	---	---	---	---
Inorganics			SW846-6010	SW846-6010	SW846-6020	SW846-6020				
Aluminum	7429-90-5	SW846-6020A	5550	50000	78789	262613	---	---	---	---
Antimony	7440-36-0	SW846-6020A	797	2500	165	667	---	---	---	---
Arsenic	7440-38-2	SW846-6020A	1050	5000	264	879	---	---	---	---
Barium	7440-39-3	SW846-6020A	150	500	229	762	---	---	---	---
Cadmium	7440-43-9	SW846-6020A	133	500	97	667	---	---	---	---
Chromium (total)	7440-47-3	SW846-6020A	278	1000	1338	4459	---	---	---	---
Copper	7440-50-8	SW846-6020A	821	2500	393	1309	---	---	---	---
Cyanide, total	57-12-5	SW846-9012A	22.3	100.00	120.0	400.00	---	---	---	---
Cyanide, available	57-12-5	OIA-1677	---	---	6.6	40	---	---	---	---
Iron	7439-89-6	SW846-6020A	1550	10000	39297	166675	---	---	---	---
Lead	7439-92-1	SW846-6020A	433	1300	181	667	---	---	---	---
Manganese	7439-96-5	SW846-6020A	256	1000	814	2713	---	---	---	---
Mercury	7439-97-6	SW846-7471A/B	---	---	11.0000	37.0000	---	---	---	---
Nickel	7440-2-0	SW846-6020A	231	1000	263	877	---	---	---	---
Selenium	7782-49-2	SW846-6020A	1110	5000	182	667	---	---	---	---
Silver	7440-22-4	SW846-6020A	344	1000	95	333	---	---	---	---
Vanadium	7440-62-2	SW846-6020A	223	1000	428	1428	---	---	---	---
Zinc	7440-66-6	SW846-6020A	933	4000	3650	12167	---	---	---	---
Other					SW846 8321	SW846 8321				
Black carbon (Gustafsson)	---	Gustafsson Method	---	---	---	---	---	---	---	---
Alkalinity	3812-32-6	SM 2320B	---	---	---	---	---	---	---	---
Ammonia	7644-41-7	EPA Method 350.1	7500	15000	---	---	---	---	---	---
Biological Oxygen Demand (BOD)	---	SM 5210B	---	---	---	---	---	---	---	---
British Therman Unit (BTU)	---	ASTM D240	---	---	---	---	---	---	---	---
Bulk Density	---	ASTM D2937	---	---	---	---	---	---	---	---
Chloride	16887-00-6	EPA Method 325.2	20000	40000	---	---	---	---	---	---
Chemical Oxygen Demand (COD)	---	EPA Method 410.4	---	---	---	---	---	---	---	---
Cyanide, Reactive	57-12-5	SW846 7.3.3.2	---	---	---	---	---	---	---	---
Flash Point	---	EPA Method 1010/1020	---	---	---	---	---	---	---	---
Fluoride	16984-48-8	SM 4500F-C	2000	4000	---	---	---	---	---	---
Free Liquids (Paint Filter)	---	SW846 9095B	---	---	---	---	---	---	---	---
Moisture Content	---	SW846 3550C	0.10 %w/w	0.10 %w/w	---	---	---	---	---	---
Nitrate/Nitrite	14797-55-8 &14797-65-0	EPA Method 353.2	971	3240	---	---	---	---	---	---
Total Kjeldahl Nitrogen	7727-37-9	SM 4500-NH3 G	21936	73120	---	---	---	---	---	---
pH	---	SW846 9045D	0.01 SU	0.10 SU	---	---	---	---	---	---

Project Compound List ^B	CAS Number	Analytical Method Name/Number ^C	Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Analytical Lab Network - Lowest Achievable Limits		Pace Grand Rapids Lab Only - Lowest Achievable Limits	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)	µg/kg (dry wt.)
Phenolics	Multiple	SW846 9066	---	---	---	---	---	---	---	---
Phosphate	14265-44-2	EPA Method 365.1	---	---	---	---	---	---	---	---
Total Phosphorus	7723-14-0	EPA Method 365.1	20000	40000	---	---	---	---	---	---
Specific Gravity	---	ASTM D5057	---	---	---	---	---	---	---	---
Sulfate	14808-79-8	SM 4500-SO4 E	20000	40000	---	---	---	---	---	---
Sulfur	7704-34-9	SW846 6010	1840	25000	---	---	---	---	---	---
Sulfur, Reactive	7704-34-10	SW846 7.3.4.2	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-0	ASTM-2974-00	---	---	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-1	Lloyd Kahn Method	33880	10000	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-2	Walkley Black Method	193270	644200	---	---	---	---	---	---
Total Organic Carbon (TOC)	7440-44-3	SW846 9060M	193970	646600	---	---	---	---	---	---
Oil and Grease	---	SW846-9071B	31800	250000	---	---	---	---	---	---
Pentachlorophenol	87-86-5	SW846-8151	14	25	2	80	---	---	---	---
Alternate Methods Provided										
Cyanide, total	57-12-5	SW846 9014	22.3	100	---	---	---	---	---	---
Chloride	16887-00-6	EPA Method 300.0 / SW846 9056A	20000	40000	---	---	---	---	---	---
Fluoride	16984-48-8	EPA Method 300.0 / SW846 9056A	2000	4000	---	---	---	---	---	---
Moisture Content	---	ASTM D2974-87	0.10 %w/w	0.10 %w/w	---	---	---	---	---	---
Total Kjeldahl Nitrogen	7727-37-9	EPA Method 351.2	21936	73120	---	---	---	---	---	---
pH	---	SW846-9045C	0.01 SU	0.10 SU	---	---	---	---	---	---
Total Phosphorous	7723-14-0	EPA Method 365.4	20000	40000	---	---	---	---	---	---
Sulfate	14808-79-8	EPA Method 300.0 / SW846 9056A	20000	40000	---	---	---	---	---	---

O: SLM C: SSW F: SLM

Notes:
ASTM = ASTM International
CAS = Chemical Abstracts Service
CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
dry wt. = dry weight
MDL = Method Detection Limit
MGP = Manufactured Gas Plants
OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
PAH = polycyclic aromatic hydrocarbon
PCB = polychlorinated biphenyl
RAF = Multi-Site Risk Assessment Framework
RCRA = Resource Conservation and Recovery Act
RL = Reporting Limit
RSK-175: RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP = Standard operating procedure
SU= Standard Unit (pH is dimensionless)
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
TPH= Total Petroleum Hydrocarbons
USEPA or EPA = United States Environmental Protection Agency
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
µg/kg = micrograms per kilogram
%w/w= percent concentration weight over weight ration
--- = Lab does not offer analysis, analysis was not requested, no data
Bold Underline Text = Lab will provide an alternate method than what is identified in method column

^A Prior to any project-specific field data collection, the selected lab's ability to achieve approved project-specific screening levels, as provided in the current RAF Addendum, will be verified.

^B The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for soil and sediment. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^C Analytical method names/numbers refer to names of analytical procedures. See lab SOPs for specific procedures.

Table 3. Multi-Site Program - Air/Soil Gas/Soil Vapor Matrix
Analytical Methods, Method Detection Limits (MDL) and Reporting Limits (RL)^A
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Project Compound List ^B	CAS Number	Analytical Method Name/Number	Pace Analytical		Test America		STAT		Eurofins	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS
Method TO-3 - VOCs			ppmv	ppmv					ppmv	ppmv
Benzene	71-43-2	TO-3/TO-3 Mod or 40 CFR 60 Appendix A	0.186	0.5	---	---	---	---	0.00023	0.0010
EPA Method 3C - Indicator Parameters			%	%	%v/v	%v/v	mol%	mol%	% volume	% volume
Methane	74-82-8	EPA Method 3C, ASTM D-1946, TO-3 Mod, or 40 CFR 60 Appendix A	0.423	1	0.024	0.04	0.013	0.05	0.000019	0.00010
Carbon dioxide	124-38-9	EPA Method 3C	0.343	2.0	0.00087	0.05	0.087	0.04	0.0010	0.010
Oxygen	7782-44-7	EPA Method 3C	0.0518	2.0	0.018	0.05	0.014	0.4	0.0029	0.10
Nitrogen	7727-37-9	EPA Method 3C	0.0518	8.0	0.067	0.5	0.03	1	0.0022	0.10
Carbon monoxide	630-0-80	EPA Method 3C	0.0356	0.4	0.013	0.1	---	1	0.0013	0.010
Method PM-10 40CFR50 Appendix J - PM-10						g		µg/m3		µg*
PM-10/Particulate Matter	---	PM-10 40CFR50 Appendix J	---	---	---	0.000500	---	1000	---	1000
Method TO-13A - PAHs					µg/m3	µg/m3	µg/PUF	µg/PUF	µg*	µg*
Naphthalene	91-20-3	TO-13A/TO-13A SIM	---	---	0.160	2.50	0.0647	1	0.78	1.0
Acenaphthylene	208-96-8	TO-13A/TO-13A SIM	---	---	0.120	2.50	0.0299	1	0.17	1.0
Acenaphthene	83-32-9	TO-13A/TO-13A SIM	---	---	0.280	2.50	0.0299	1	0.16	1.0
Fluorene	86-73-7	TO-13A/TO-13A SIM	---	---	0.170	2.50	0.03143	1	0.19	1.0
Anthracene	120-12-7	TO-13A/TO-13A SIM	---	---	0.170	2.50	0.01534	1	0.18	1.0
Phenanthrene	85-01-8	TO-13A/TO-13A SIM	---	---	0.250	2.50	0.02169	1	0.16	1.0
Fluoranthene	206-44-0	TO-13A/TO-13A SIM	---	---	0.120	2.50	0.02376	1	0.20	1.0
Pyrene	129-00-0	TO-13A/TO-13A SIM	---	---	0.170	2.50	0.03497	1	0.18	1.0
Benz(a)anthracene	56-55-3	TO-13A/TO-13A SIM	---	---	0.110	2.50	0.02376	1	0.18	1.0
Chrysene	218-01-9	TO-13A/TO-13A SIM	---	---	0.160	2.50	0.0394	1	0.20	1.0
Benzo(b)fluoranthene	205-99-2	TO-13A/TO-13A SIM	---	---	0.230	2.50	0.03819	1	0.27	1.0
Benzo(k)fluoranthene	207-08-9	TO-13A/TO-13A SIM	---	---	0.200	2.50	0.03143	1	0.24	1.0
Benzo(a)pyrene	50-32-8	TO-13A/TO-13A SIM	---	---	0.130	2.50	---	---	0.19	1.0
Benzo(g,h,i)perylene	191-24-2	TO-13A/TO-13A SIM	---	---	0.270	2.50	0.02169	1	0.31	1.0
Indeno(1,2,3-cd)pyrene	193-39-5	TO-13A/TO-13A SIM	---	---	0.150	2.50	0.02376	1	0.34	1.0
Dibenz(a,h)anthracene	53-70-3	TO-13A/TO-13A SIM	---	---	0.220	2.50	0.02169	1	0.27	1.0

Project Compound List ^B	CAS Number	Analytical Method Name/Number	Pace Analytical		Test America		STAT		Eurofins	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS
Method TO-14 - VOCs and Naphthalene ^C					ppb v/v	ppb v/v	ppbv	ppbv	µg/m3	µg/m3
1,1,2,2-Tetrachloroethane	79-34-5	TO-14	---	---	0.034	0.2	0.013759	0.2	0.53	3.4
1,1,2-Trichloroethane (Vinyl trichloride)	79-00-5	TO-14	---	---	0.037	0.2	0.019325	0.2	0.85	2.7
1,1-Dichloroethane (Ethylidene chloride)	74-34-3	TO-14	---	---	0.028	0.2	0.009709	0.2	0.52	2.0
1,2,4-Trichlorobenzene	120-82-1	TO-14	---	---	0.034	2	0.044273	0.2	1.3	15
1,2,4-Trimethylbenzene (Pseudocumene)	95-63-6	TO-14	---	---	0.016	0.2	0.018122	0.2	0.28	2.4
1,2-Dibromoethane (Ethylene dibromide)	106-93-4	TO-14	---	---	0.018	0.2	0.022381	0.2	0.47	3.8
1,2-Dichloroethane (Ethylene dichloride)	107-06-2	TO-14	---	---	0.052	0.2	0.02305	0.2	0.63	2.0
1,2-Dichloropropane (Propylene dichloride)	78-87-5	TO-14	---	---	0.035	0.2	0.013679	0.2	0.53	2.3
1,3,5-Trimethylbenzene (Mesitylene)	108-67-8	TO-14	---	---	0.019	0.2	0.013784	0.2	0.41	2.4
Benzene (Cyclohexatriene)	71-43-2	TO-14	---	---	0.029	0.2	0.014333	0.2	0.31	1.6
Benzyl chloride (cx-Chlorotoluene)	100-44-7	TO-14	---	---	0.018	0.8	0.2 ppbv	0.5	0.43	3.0
Carbon tetrachloride (Tetrachloromethane)	56-23-5	TO-14	---	---	0.011	0.2	0.027957	0.2	0.75	3.1
Chlorobenzene (Phenyl chloride)	108-90-7	TO-14	---	---	0.018	0.2	0.012641	0.2	0.42	2.3
Chloroform (Trichloromethane)	67-66-3	TO-14	---	---	0.038	0.2	0.010978	0.2	0.33	2.4
cis-1,2-Dichloroethylene	156-59-2	TO-14	---	---	0.03	0.2	0.014721	0.2	0.71	2.0
cis-1,3-Dichloropropene (cis-1,3- dichloropropylene)	542-75-6	TO-14	---	---	0.029	0.2	0.023468	0.2	0.61	2.3
Dichloromethane (Methylene chloride)	75-09-2	TO-14	---	---	0.12	0.5	0.107600	0.2	1.0	17
Ethyl chloride (Chloroethane)	75-00-3	TO-14	---	---	0.061	0.8	0.2	0.2	1.1	5.3
Ethylbenzene	100-41-4	TO-14	---	---	0.02	0.2	0.015603	0.2	0.68	2.2
Freon 11 (Trichlorofluoromethane)	75-69-4	TO-14	---	---	0.045	0.2	0.012616	0.2	0.42	2.8
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	76-13-1	TO-14	---	---	0.041	0.2	0.007505	0.2	1.1	3.8
Freon 114 (1,2-Dichloro-1,1,2,2- tetrafluoroethane)	76-14-2	TO-14	---	---	0.052	0.2	0.028791	1	0.92	3.5
Freon 12 (Dichlorodifluoromethane)	75-71-8	TO-14	---	---	0.056	0.5	0.006492	0.2	0.54	2.5
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro- 1,3-butadiene)	87-68-3	TO-14	---	---	0.036	2	0.022554	0.2	3.2	21
m-Dichlorobenzene (1,3-Dichlorobenzene)	541-73-1	TO-14	---	---	0.02	0.2	0.016922	0.2	0.74	3.0
Methyl bromide (Bromomethane)	74-83-9	TO-14	---	---	0.044	0.2	0.02337	0.5	1.1	19
Methyl chloride (Chloromethane)	74-87-3	TO-14	---	---	0.06	0.5	0.049459	0.2	1.2	10
Methyl chloroform (1,1,1-Trichloroethane)	71-55-6	TO-14	---	---	0.03	0.2	0.010498	0.2	0.46	2.7
m-Xylene (1,3-Dimethylbenzene)/m,p-xyelene	108-38-3	TO-14	---	---	0.025	0.8	0.029472	0.4	0.62	2.2
Naphthalene	91-20-3	TO-14	---	---	0.03	0.5	---	---	0.22	5.2
o-Dichlorobenzene (1,2-dichlorobenzene)	95-50-1	TO-14	---	---	0.018	0.2	0.016647	0.2	0.41	3.0
o-Xylene (1,2-Dimethylbenzene)	95-47-6	TO-14	---	---	0.018	0.2	0.012733	0.2	0.83	2.2
p-Dichlorobenzene (1,4-dichlorobenzene)	106-46-7	TO-14	---	---	0.019	0.2	0.021167	0.2	0.43	3.0
p-Xylene (,14-Dimethylxylene)	106-42-3	TO-14	---	---	0.025	0.8	0.029472	0.4	---	---

Table 3

Project Compound List ^B	CAS Number	Analytical Method Name/Number	Pace Analytical		Test America		STAT		Eurofins	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS
Styrene (Vinyl benzene)	100-42-5	TO-14	---	---	0.016	0.2	0.021273	0.2	0.35	2.1
Tetrachloroethylene (Perchloroethylene)	127-18-4	TO-14	---	---	0.03	0.2	0.014467	0.2	1.1	3.4
Toluene (Methyl benzene)	108-88-3	TO-14	---	---	0.025	0.2	0.022569	0.2	0.36	1.9
trans-1,3-Dichloropropene (trans-1,3-Dichloropropylene)	542-75-6	TO-14	---	---	0.026	0.2	0.0272	0.2	0.60	2.3
Trichloroethylene (Trichloroethene)	79-01-6	TO-14	---	---	0.03	0.2	0.015249	0.2	0.86	2.7
Vinyl chloride (Chloroethylene)	75-01-4	TO-14	---	---	0.026	0.2	0.016859	0.2	0.36	1.3
Vinylidene chloride (1,1-Dichloroethene)	75-35-4	TO-14	---	---	0.01	0.2	0.012609	0.2	0.85	2.0
Method TO-15 - VOCs and Naphthalene			µg/m3	µg/m3	ppb v/v	ppb v/v	ppbv	ppbv	µg/m3	µg/m3
1,1,2-Trichloroethane; C2H3Cl3	79-00-5	TO-15	0.225	0.555	0.037	0.2	0.025368	0.2	0.85	2.7
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1	TO-15	0.958	3.77	0.034	2	0.03801	0.2	1.3	15
1,3-Butadiene; C4H6	106-99-0	TO-15	0.206	0.450	0.037	0.2	0.046824	0.2	0.35	1.1
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6	TO-15	---	---	0.029	0.2	0.023786	0.2	0.61	2.3
1,4-Dichlorobenzene (p-); C6H4Cl2	106-46-7	TO-15	0.219	1.22	0.019	0.2	0.017137	0.2	0.43	3
1,4-Dioxane (1,4-Diethylene oxide); C4H8O2	123-91-1	TO-15	0.354	3.66	0.16	5	0.079633	0.5	1.8	7.2
2,2,4-Trimethyl pentane C8H18	540-84-1	TO-15	0.432	2.37	0.043	0.2	---	---	0.33	2.3
Acetonitrile (cyanomethane); C2H3N	75-05-8	TO-15	---	---	1.1	5	---	---	---	---
Acrolein (2-propenal); C3H4O	107-02-8	TO-15	0.256	1.17	1.5	5	---	---	---	---
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1	TO-15	0.326	1.10	0.18	0.5	---	---	---	---
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1	TO-15	0.363	1.59	0.063	0.5	---	---	---	---
Benzene; C6H6	71-43-2	TO-15	0.151	0.325	0.029	0.2	0.016665	0.2	0.31	1.6
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7	TO-15	0.236	1.05	0.018	0.8	0.06071	0.5	0.43	3.0
Bromoform (tribromomethane); CHBr3	75-25-2	TO-15	0.691	2.10	0.025	0.2	0.009746	0.5	0.64	5.2
Carbon disulfide; CS2	75-15-0	TO-15	0.179	0.633	0.03	0.5	0.02054	0.2	0.69	6.2
Carbon tetrachloride; CCl4	56-23-5	TO-15	0.318	0.639	0.011	0.2	0.044972	0.2	0.75	3.1
Chlorobenzene; C6H5Cl	108-90-7	TO-15	0.179	0.936	0.018	0.2	0.05	0.2	0.42	2.3
Chloroform; CHCl3	67-66-3	TO-15	0.231	0.496	0.038	0.2	0.010978	0.2	0.33	2.4
Cumene (isopropylbenzene); C9H12	98-82-8	TO-15	0.330	2.50	0.019	0.8	---	---	0.33	2.4
Ethyl chloride (chloroethane); C2H5Cl	75-00-3	TO-15	0.204	0.536	0.061	0.8	0.04054	0.2	1.1	5.3
Ethylbenzene; C8H10	100-41-4	TO-15	0.171	0.883	0.02	0.2	0.014536	0.2	0.68	2.2
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4	TO-15	0.333	1.56	0.018	0.2	0.021725	0.2	0.47	3.8
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2	TO-15	0.198	0.411	0.052	0.2	0.023117	0.2	0.63	2.0
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75-34-3	TO-15	0.212	0.823	0.028	0.2	0.008674	0.2	0.52	2.0
Hexachlorobutadiene; C4Cl6	87-68-3	TO-15	0.869	2.17	0.036	2	0.020767	0.2	3.2	21
Hexane; C6H14	110-54-3	TO-15	0.333	0.716	0.028	0.8	0.035056	0.5	0.50	1.0
1,1,2,2-Tetrachloroethane; C2H2Cl4	79-34-5	TO-15	0.290	0.698	0.028	0.8	0.013758	0.2	0.53	3.4

Table 3

Project Compound List ^B	CAS Number	Analytical Method Name/Number	Pace Analytical		Test America		STAT		Eurofins	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS
Methyl bromide (bromomethane); CH ₃ Br	74-83-9	TO-15	0.208	0.789	0.044	0.2	0.02337	0.5	1.1	19
Methyl chloride (chloromethane); CH ₃ Cl	74-87-3	TO-15	0.134	0.420	0.06	0.5	0.113286	0.5	1.2	10
Methyl chloroform (1,1,1-trichloroethane); C ₂ H ₃ Cl ₃	71-55-6	TO-15	0.342	1.11	0.03	0.2	---	---	0.46	2.7
Methyl ethyl ketone (2-butanone); C ₄ H ₈ O	78-93-3	TO-15	0.203	3.00	0.092	1	0.082803	0.5	1.4	5.9
Methyl isobutyl ketone (hexone); C ₆ H ₁₂ O	108-10-1	TO-15	0.356	4.16	0.18	0.5	---	---	0.66	2.0
Methyl methacrylate; C ₅ H ₈ O ₂	80-62-6	TO-15	0.365	0.832	---	---	---	---	---	---
Methyl tert-butyl ether; C ₅ H ₁₂ O	1634-04-4	TO-15	0.667	3.66	0.022	1	0.011323	0.2	0.94	1.8
Methylene chloride; CH ₂ Cl ₂	75-09-2	TO-15	1.52	3.53	0.12	0.5	0.199322	2	1.0	17
m-Xylene; C ₈ H ₁₀ /m,p-xylene	108-38-3	TO-15	0.349	1.77	0.025	0.8	0.024937	0.4	0.62	2.2
Naphthalene	91-20-3	TO-15	0.597	2.66	0.03	0.5	0.057118	0.2	0.22	5.2
o-Xylene; C ₈ H ₁₀	95-47-6	TO-15	0.371	0.883	0.018	0.2	0.012733	0.2	0.83	2.2
Propylene dichloride (1,2-dichloropropane); C ₃ H ₆ Cl ₂	78-87-5	TO-15	0.306	0.939	0.035	0.2	0.037069	0.2	0.53	2.3
p-Xylene; C ₈ H ₁₀ /m,p-xylene	106-42-3	TO-15	0.349	1.77	0.025	0.8	0.024937	0.4	---	---
Styrene; C ₈ H ₈	100-42-5	TO-15	0.167	0.866	0.016	0.2	0.047119	0.2	0.35	2.1
Tetrachloroethylene; C ₂ Cl ₄	127-18-4	TO-15	0.287	0.689	0.03	0.2	0.014467	0.2	1.1	3.4
Toluene; C ₇ H ₈	108-88-3	TO-15	0.159	0.766	0.025	0.2	0.022569	0.2	0.36	1.9
Trichloroethylene; C ₂ HCl ₃	79-01-6	TO-15	0.268	0.546	0.03	0.2	0.015249	0.2	0.86	2.7
Vinyl acetate; C ₄ H ₆ O ₂	108-05-4	TO-15	0.166	0.716	0.083	5	0.080086	2	---	---
Vinyl bromide (bromoethene); C ₂ H ₃ Br	593-60-2	TO-15	0.230	4.45	0.02	0.2	---	---	---	---
Vinyl chloride (chloroethene); C ₂ H ₃ Cl	75-01-4	TO-15	0.126	0.260	0.026	0.2	0.016859	0.2	0.36	1.3
Vinylidene chloride (1,1-dichloroethylene); C ₂ H ₂ Cl ₂	75-35-4	TO-15	0.237	0.806	0.01	0.2	0.012609	0.2	0.85	2.0
Xylenes (isomer & mixtures); C ₈ H ₁₀	1330-20-7	TO-15	0.371	2.65	0.04	0.7	0.03702	0.6	---	---
Method TO-17 - VOCs and PAHs										
1,1,1,2-Tetrachloroethane	630-20-6	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1,1,1-Trichloroethane	71-55-6	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1,1,2,2-Tetrachloroethane	79-34-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1,1,2-Trichloroethane	79-00-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1,2,3-Trimethylbenzene	526-73-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1,2-Dichloroethane	107-06-2	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1,3,5-Trimethylbenzene	108-67-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
1-Methyl-3-ethylbenzene	620-14-4	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
3,5,5-Trimethylcyclohex-2-enone	78-59-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Acetic acid	64-1-97	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Acetone	67-64-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D

Table 3

Project Compound List ^B	CAS Number	Analytical Method Name/Number	Pace Analytical		Test America		STAT		Eurofins	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS
Acetonitrile	75-05-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Acrylonitrile	107-13-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
all Xylenes	1330-2-07	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Aniline	62-53-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Benzene	71-43-2	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Butoxyethanol	111-7-62	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Butoxyethylacetate	124-1-74	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Butylacetate	123-86-4	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Carbontetrachloride	56-23-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Chlorobenzene	108-90-7	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Cyclohexanone	108-94-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Decane	124-18-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Dichloromethane	75-09-2	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Ethanol	64-17-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Ethoxyethanol	7518-70-9	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Ethoxyethylacetate	817-95-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Ethylacetate	141-7-86	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Ethylacrylate	140-88-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Ethylbenzene	100-41-4	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Furfural	98-01-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
iso-Butanol	78-83-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Isobutylacetate	110-1-90	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Isopropanol	67-63-0	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Isopropylacetate	108-2-14	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Isopropylbenzene	98-82-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Maleic anhydride	108-31-6	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methanol	67-56-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methoxyethanol	111-7-73	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methoxyethylacetate	3938-96-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methoxypropanol	13071-62-0	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methyl-2-ethylbenzene	611-1-43	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methyl-4-ethylbenzene	622-9-68	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methylacetate	79-2-09	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methylacrylate	96-33-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methylethylketone (2-butanone)	78-9-33	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methylisobutylketone	108-1-01	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methylmethacrylate	80-6-26	TO-17	---	---	---	---	---	---	see footnote D	see footnote D

Table 3

Project Compound List ^B	CAS Number	Analytical Method Name/Number	Pace Analytical		Test America		STAT		Eurofins	
			MDL	RL	MDL	RL	MDL	RL	MDL	RL
			UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS	UNITS
Methylstyrene	98-83-9	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Methyl-t-butyl ether	1634-0-44	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Butanal	123-72-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Butane	106-97-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Butanol	71-36-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Dodecane	112-4-03	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Heptane	142-82-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Hexane	110-54-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Nitrobenzene	98-95-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Nonane	111-84-2	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Octane	111-6-59	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Pentane	109-6-60	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Propanol	71-23-8	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Propylbenzene	103-65-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
n-Undecane	1120-2-14	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Octanol	111-8-75	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Phenol	108-95-2	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Propionitrile	107-12-0	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Propylacetate	109-6-04	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Pyridine	110-86-1	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Styrene	100-42-5	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
t-Butylacetate	540-8-85	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Tetrachloroethylene	127-18-4	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Toluene	108-88-3	TO-17	---	---	---	---	---	---	see footnote D	see footnote D
Trichloroethylene	79-01-6	TO-17	---	---	---	---	---	---	see footnote D	see footnote D

O: SLM C: SSW F: SLM

Notes:
ASTM = ASTM International
CAS = Chemical Abstracts Service
CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act
CFR= Code of Federal Regulations
g = gram
m³ = cubic meter
MDL= Method Detection Limit
MGP= Manufactured gas plant
mol% = mole percent

PAH = polycyclic aromatic hydrocarbon
PM₁₀ = particulate matter with a nominal diameter of 10 micrometers or less.
ppbv = parts per billion by volume
ppb v/v = part per billion volume/volume ratio
ppmv = parts per million by volume
PUF = polyurethane foam
RAF= Multi-Site Risk Assessment Framework
RL= Reporting Limit
SIM = selective ion monitoring
SOP= standard operating procedure
TO-3 = USEPA Method TO-3, Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection. See lab SOPs for additional details.
TO-13A = USEPA Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons in Ambient Air Using Gas Chromatography/Mass Spectrometry. See lab SOPs for additional details.

TO-14 = USEPA Compendium Method TO-14A, Determination Of Volatile Organic Compounds In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography. See lab SOPs for additional details.
TO-15 = USEPA Compendium method TO-15, Determination Of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry. See lab SOPs for additional details.
TO-17 = USEPA Method TO-17, Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. See lab SOPs for additional details.
USEPA or EPA = United States Environmental Protection Agency
VOC = volatile organic compound
µg = microgram
%v/v = percent concentration volume/volume ratio
--- = Lab does not offer analysis, analysis was not requested, no data

* Note from lab: The instrument units are in mass; the sample volume collected will dictate concentration. For PAHs, a 200 cubic meter volume would result in RLs of 0.005 ug/m³ and for PM₁₀ a 200 cubic meter volume sample will result in a concentration of 5 ug/m³.

^A The list of compounds provided are those expected to be used for the MGP Multi-Site Program, as identified in the RAF (2007) and RAF Addenda, and some common additional compounds/analyses. The RAF identifies the sources of screening levels for soil and sediment. Other project-specific analytes of interest will be listed in Site-Specific Work Plans.

^B Other compounds/analytes may be identified in Site-Specific Work Plans.

^C Eurofins does not analyze samples using TO-14 methodology, but uses TO-15 methodology for TO-14 analytical requests. TO-15 is the newer air method for VOC analysis and meets the TO-14 requirements. Test America also prefers TO-15. Eurofins and Test America responses in this section are for TO-15.

^D TO-17 RLs and MDLs vary based on the sorbent tube used (VI vs. Tenax). Additionally, the RL and MDL in ug/m³ changes depending on the amount sampled through the tubes. RLs and MDLs will be determined on a project-specific basis prior to field data collection.

Table 4A. Sampling and Analysis Summary - Alpha
Water Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Petroleum Volatile Organic Compounds	SW846-8260B/ 8021B	3/40 mL G TLS	HCl	40 mL	14 days
Petroleum Volatile Organic Compounds	WI DRO	2/1-L AG TLC	none ^C	1 L	7 days
Gasoline Range Organics	WI GRO	3/40 mL G TLS	HCl	40 mL	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	2/1-L AG TLC	none ^C	1 L	7 days
Semivolatile Organic Compounds	SW846-8270C/D	2/1-L AG TLC	none ^C	1 L	7 days
Phenols					
Phenols	SW846-8270C/D	2/1-L AG TLC	none ^C	1 L	7 days
Phenolics	SW846 9066				
Pesticides					
Pesticides	SW846 8081B	2/500 mL AG TLC	none ^C	500 mL	7 days
PCBs					
PCBs	SW846 8082A	2/1-L AG TLC	none ^C	1 L	7 days
Inorganics					
Metals, total	SW846-6020A/ 6010	1/250 mL P	HNO ₃	250 mL	180 days
Metals, dissolved	SW846-6020A/ 6010	1/250 mL P	HNO ₃ after filtration	250 mL	180 days
Cyanide, total	SW846-9012A	1/250 mL P	NaOH	250 mL	14 days
Cyanide, available	OIA-1677	1/250 mL P	NaOH	250 mL	14 days
Cyanide, amenable	SW846 9012B/9014	1/500 mL P	NaOH	500 mL	14 days
Cyanide, dissociable	SM 4500CN	1/250 mL P	NaOH	250 mL	14 days
Mercury	SW846-7471B	1/500 mL P	HNO ₃	500 mL	28 days
Other					
Alkalinity as CaCO ₃	SM 2320B	1/120 mL P	none ^C	120 mL	14 days
Ammonia	EPA Method 350.1	1/500 mL P	H ₂ SO ₄	500 mL	28 days
Biochemical Oxygen Demand (BOD)	SM 5210B	1/950 mL P	none ^C	950 mL	48 hours
Chloride	SM 4500-Cl E	1/250 mL P	none ^C	250 mL	28 days
Chemical Oxygen Demand (COD)	EPA Method 410.4	1/120 mL P	H ₂ SO ₄	120 mL	28 days
Fluoride	SM 4500F-C	1/500 mL P	none ^C	500 mL	28 days
Hardness	SW846 6010	1/500 mL P	HNO ₃	500 mL	180 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Methane	RSK-175	1/20 mL G	HCl or H ₂ SO ₄	20 mL	14 days
Nitrate	EPA Method 353.2	1/250 mL P	none ^C	250 mL	48 hours
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	1/250 mL P	H ₂ SO ₄	250 mL	28 days
Oil & Grease	EPA Method 1664A	2/1-L AG TLC	HCl	1 L	28 days
pH	EPA Method 150.1/ SM 9040/ SM 9045	1/250 mL P	none ^C	250 mL	24 hours
Phosphate	EPA Method 365.1	1/500 mL P	H ₂ SO ₄	500 mL	28 days
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	1/950 mL P	none ^C	950 mL	7 days
Sulfate	SM 4500 SO ₄ E	1/500 mL P	none ^C	500 mL	28 days
Sulfur	SW846 6010	1/500 mL P	HNO ₃	500 mL	180 days
Pentachlorophenol	SW846-8151	2/1-L AG TLC	none ^C	1 L	7 days
Total Organic Carbon (TOC)	SW846 9060	3/40 mL G TLS	H ₂ SO ₄	40 mL	28 days
Alternate Methods Provided					
Hardness	SM 2340B	1/500 mL P	HNO ₃	500 mL	180 days
Phosphate	SM 4500P-E	1/500 mL P	H ₂ SO ₄	500 mL	28 days
1,2-Dibromo-3-chloropropane (DBCP)	SW846 8260C	3/40 mL G TLS	HCL	40 mL	14 days
Hexachlorocyclopentadiene	SW846 8270D	2/1-L AG TLC	none ^C	1-L	7 days

O: SLM

C: SSW

F: SLM

Notes:

AG= Amber Glass

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G = glass

L = liter

MGP=Manufactured Gas Plant

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

P = poly

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

TLC = Teflon-lined cap

TLS = Teflon-lined septum

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4A. Sampling and Analysis Summary - Brighton

Water Matrix

MGP Multi-Site Program

USEPA Region 5

CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Petroleum Volatile Organic Compounds	SW846-8260B/ 8021B	40 mL VOA vial	HCl	2-40 mL VOA vials	14 days
Petroleum Volatile Organic Compounds	WI DRO	40 mL VOA vial	HCl	2-40 mL VOA vials	14 days
Gasoline Range Organics	WI GRO	40 mL VOA vial	HCl	2-40 mL VOA vials	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	1 Liter AG	None ^C	1 Liter	7 days
Semivolatile Organic Compounds	SW846-8270C/D	1 Liter AG	None ^C	1 Liter	7 days
Phenols					
Phenols	SW846-8270C/D	1 Liter AG	None ^C	1 Liter	7 days
Phenolics	SW846 9066	250mL HDPE	H ₂ SO ₄	250 mL	28 days
Pesticides					
Pesticides	SW846 8081B	1 Liter AG	None ^C	1 Liter	7 days
PCBs					
PCBs	SW846 8082A	1 Liter AG	None ^C	1 Liter	7 days
Inorganics					
Metals, total	SW846-6020A/ 6010	250 mL HDPE	HNO ₃	250 mL	6 months
Metals, dissolved	SW846-6020A/ 6010	250 mL HDPE	HNO ₃	250 mL	6 months
Cyanide, total	SW846-9012A	125 mL HDPE	NaOH	125 mL	14 days
Cyanide, available	OIA-1677	125 mL HDPE	NaOH	125 mL	14 days
Cyanide, amenable	SW846 9012B/9014	125 mL HDPE	NaOH	125 mL	14 days
Cyanide, dissociable	SM 4500CN				
Mercury	SW846-7471B	250 mL HDPE	HNO ₃	250 mL	28 days
Other					
Alkalinity as CaCO ₃	SM 2320B	500 mL HDPE	None ^C	500 mL	14 days
Ammonia	EPA Method 350.1	250 mL HDPE	H ₂ SO ₄	250 mL	28 days
Biochemical Oxygen Demand (BOD)	SM 5210B	500 mL HDPE	None ^C	500 mL	48 hours
Chloride	SM 4500-Cl E	500 mL HDPE	None ^C	500mL	28 days
Chemical Oxygen Demand (COD)	EPA Method 410.4	250 mL HDPE	H ₂ SO ₄	250 mL	28 days
Fluoride	SM 4500F-C	500 mL HDPE	None ^C	500mL	28 days
Hardness	SW846 6010	250 mL HDPE	HNO ₃	250 mL	6 months
Methane	RSK-175	40 mL VOA vial	None ^C	40 mL VOA vial	7 days
Nitrate	EPA Method 353.2	500 mL HDPE	None ^C	500 mL	48 hours

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	500 mL HDPE	None ^C	500 mL	48 hours
Oil & Grease	EPA Method 1664A	250 mL AG	H ₂ SO ₄	250 mL	28 days
pH	EPA Method 150.1/ SM 9040/ SM 9045	500 mL HDPE	None ^C	500 mL	as soon as possible
Phosphate	EPA Method 365.1	500 mL HDPE	None ^C	500 mL	48 hours
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	500 mL HDPE	None ^C	500 mL	7 days
Sulfate	SM 4500 SO ₄ E	500 mL HDPE	None ^C	500 mL	28 days
Sulfur	SW846 6010				
Pentachlorophenol	SW846-8151	1 Liter AG	None ^C	1 Liter	1 week
Total Organic Carbon (TOC)	SW846 9060	40 mL VOA vial	HCl	1 40mL VOA Vial	28 days
Alternate Methods Provided					
Hardness	SM 2340B	250 mL HDPE	HNO ₃	250 mL	6 months
Phosphate	SM 4500P-E	500 mL HDPE	None ^C	500 mL	48 hours

O: SLM

C: SSW

F: SLM

Notes:

AG= Amber Glass

C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

HDPE = high density polyethylene

MGP= Manufactured Gas Plants

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

USEPA or EPA= United States Environmental Protection Agency

VOA = volatile organic analysis

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4A. Sampling and Analysis Summary - STAT

Water Matrix

MGP Multi-Site Program

USEPA Region 5

CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Petroleum Volatile Organic Compounds	SW846-8260B/ 8021B	40mL Glass VOA	HCl	40mL	14 Days
Petroleum Volatile Organic Compounds	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	1L Amber Glass	Cool 4°C	500 mL	7 Days
Semivolatile Organic Compounds	SW846-8270C/D	1L Amber Glass	Cool 4°C	500 mL	7 Days
Phenols					
Phenols	SW846-8270C/D	1L Amber Glass	Cool 4°C	500 mL	7 Days
Phenolics	SW846 9066	500 Amber Glass	H2SO4, Cool 4°C	100 mL	28 Days
Pesticides					
Pesticides	SW846 8081B	1L Amber Glass	Cool 4°C	500 mL	7 Days
PCBs					
PCBs	SW846 8082A	1L Amber Glass	Cool 4°C	500 mL	None
Inorganics					
Metals, total	SW846-6020A/ 6010	500mL Poly	HNO3	250 mL	180 Days
Metals, dissolved	SW846-6020A/ 6010	500mL Poly (Field Filtered)	HNO3	250 mL	180 Days
Cyanide, total	SW846-9012A	250mL Poly	NaOH, Cool 4°C	100 mL	14 Days
Cyanide, available	OIA-1677				
Cyanide, amenable	SW846 9012B/9014	250mL Poly	NaOH, Cool 4°C	100 mL	14 Days
Cyanide, dissociable	SM 4500CN	250mL Poly	NaOH, Cool 4°C	100 mL	14 Days
Mercury	SW846-7471B	250mL Poly	HNO3, Cool 4°C	100 mL	28 Days
Other					
Alkalinity as CaCO ₃	SM 2320B	1L Poly or Glass	Cool 4°C	500 mL	14 Days
Ammonia	EPA Method 350.1	500mL Amber Glass	H2SO4, Cool 4°C	250 mL	28 Days
Biochemical Oxygen Demand (BOD)	SM 5210B	950mL Poly	Cool 4°C	500 mL	48 Hours
Chloride	SM 4500-Cl E	500mL Poly	Cool 4°C	250 mL	1 Day
Chemical Oxygen Demand (COD)	EPA Method 410.4	500mL Amber Glass	H2SO4, Cool 4°C	250 mL	28 Days
Fluoride	SM 4500F-C	250mL Poly	Cool 4°C	100 mL	28 Days
Hardness	SW846 6010	500mL Poly	HNO3	250 mL	180 Days
Methane	RSK-175				
Nitrate	EPA Method 353.2	250mL Poly	Cool 4°C	100 mL	48 Hours
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	250mL Poly	H2SO4, Cool 4°C	100 mL	28 Days
Oil & Grease	EPA Method 1664A	950mL Amber Glass	H2SO4, Cool 4°C	950 mL	28 Days
pH	9045	250mL Poly	Cool 4°C	100 mL	0 Days
Phosphate	EPA Method 365.1	500mL Amber Glass	H2SO4, Cool 4°C	100 mL	28 Days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	950mL Poly	Cool 4°C	500 mL	7 Days
Sulfate	SM 4500 SO4 E	250mL Poly	Cool 4°C	100 mL	28 Days
Sulfur	SW846 6010				
Pentachlorophenol	SW846-8151	1L Amber Glass	Cool 4°C	500 mL	7 Days
Total Organic Carbon (TOC)	SW846 9060				
Alternate Methods Provided					
Hardness	SM 2340B	500mL Poly	HNO3	100 mL	180 Days
Phosphate	SM 4500P-E	500mL Amber Glass	H2SO4, Cool 4°C	100 mL	28 Days
			O: SLM	C: SSW	F: SLM

Notes:

°C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

L = liter

MGP= Manufactured Gas Plant

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition

USEPA or EPA= United States Environmental Protection Agency

VOA = volatile organic analysis

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4A. Sampling and Analysis Summary - Battelle
Water Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Petroleum Volatile Organic Compounds	SW846-8260B/ 8021B				
Petroleum Volatile Organic Compounds	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	G with TLC	Cool <6C	1L	7 days
Semivolatile Organic Compounds	SW846-8270C/D				
Phenols					
Phenols	SW846-8270C/D				
Phenolics	SW846 9066				
Pesticides					
Pesticides	SW846 8081B				
PCBs					
PCBs	SW846 8082A				
Inorganics					
Metals, total	SW846-6020A/ 6010				
Metals, dissolved	SW846-6020A/ 6010				
Cyanide, total	SW846-9012A				
Cyanide, available	OIA-1677				
Cyanide, amenable	SW846 9012B/9014				
Cyanide, dissociable	SM 4500CN				
Mercury	SW846-7471B				
Other					
Alkalinity as CaCO ₃	SM 2320B				
Ammonia	EPA Method 350.1				
Biochemical Oxygen Demand (BOD)	SM 5210B				
Chloride	SM 4500-Cl E				
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Fluoride	SM 4500F-C				
Hardness	SW846 6010				
Methane	RSK-175				
Nitrate	EPA Method 353.2				
Nitrogen, Nitrate & Nitrite	EPA Method 353.2				
Oil & Grease	EPA Method 1664A				
pH	EPA Method 150.1/ SM 9040/ SM 9045				
Phosphate	EPA Method 365.1				
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D				
Sulfate	SM 4500 SO4 E				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Sulfur	SW846 6010				
Pentachlorophenol	SW846-8151				
Total Organic Carbon (TOC)	SW846 9060				
Alternate Methods Provided					
Hardness	SM 2340B				
Phosphate	SM 4500P-E				
			O: SLM	C: SSW	F: SLM

Notes:

C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G= Glass

L = liter

MGP= Manufactured Gas Plant

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

TLC= Teflon-line cap

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

< = less than

^A For list of compounds in this analytical group, see series of Tables 1 and 2.

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4A. Sampling and Analysis Summary - ESS

Water Matrix

MGP Multi-Site Program

USEPA Region 5

CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Petroleum Volatile Organic Compounds	SW846-8260B/ 8021B	3x 40ml VOA AG ZHS	HCL	3x 40ml	14 days
Petroleum Volatile Organic Compounds	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	2x1L AG	None ^C	1x1L	7 days
Semivolatile Organic Compounds	SW846-8270C/D	2x1L AG	None ^C	1x1L	7 days
Phenols					
Phenols	SW846-8270C/D	2x1L AG	None ^C	1x1L	7 days
Phenolics	SW846 9066	1L P	H2SO4	0.5L	28 days
Pesticides					
Pesticides	SW846 8081B	2x1L AG	None ^C	1x1L	7 days
PCBs					
PCBs	SW846 8082A	2x1L AG	None ^C	1x1L	7 days
Inorganics					
Metals, total	SW846-6020A/ 6010	250ml P	HNO3	250ml	6 months
Metals, dissolved	SW846-6020A/ 6010	250ml P	HNO3 (field filtered)	250ml	6 months
Cyanide, total	SW846-9012A	250ml P	NaOH	250ml	14 days
Cyanide, available	OIA-1677	250ml P	NaOH	250ml	14 days
Cyanide, amenable	SW846 9012B/9014	250ml P	NaOH	250ml	14 days
Cyanide, dissociable	SM 4500CN	250ml P	NaOH	250ml	14 days
Mercury	SW846-7471B	250ml P	HNO3	250ml	28 days
Other					
Alkalinity as CaCO ₃	SM 2320B	250ml P	None ^C	250ml	14 days
Ammonia	EPA Method 350.1	500ml P	H2SO4	250ml	28 days
Biochemical Oxygen Demand (BOD)	SM 5210B	1L P	None ^C	1L	48 hours
Chloride	SM 4500-Cl E	250ml P	None ^C	250ml	28 days
Chemical Oxygen Demand (COD)	EPA Method 410.4	250ml P	H2SO4	250ml	28 days
Fluoride	SM 4500F-C				
Hardness	SW846 6010	250ml P	HNO3	250ml	6 months
Methane	RSK-175	3x 40ml VOA AG ZHS	HCL	3x 40ml	14 days
Nitrate	EPA Method 353.2	250ml P	None ^C	250ml	28 days
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	250ml P	H2SO4	250ml	28 days
Oil & Grease	EPA Method 1664A	1x1L AG	H2SO4	1x1L	28 days
pH	EPA Method 150.1/ SM 9040/ SM 9045	250ml P	None ^C	250ml	Immediate
Phosphate	EPA Method 365.1	500ml P	H2SO4	250ml	28 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	250ml P	None ^C	250ml	7 days
Sulfate	SM 4500 SO4 E	250ml P	None ^C	250ml	28 days
Sulfur	SW846 6010				
Pentachlorophenol	SW846-8151				
Total Organic Carbon (TOC)	SW846 9060	2x40ml VOA G	H2SO4	2x40ml	28 days
Alternate Methods Provided					
Hardness	SM 2340B	250ml P	HNO3	250ml	6 months
Phosphate	SM 4500P-E	500ml P	H2SO4	250ml	28 days
			O: SLM	C: SSW	F: SLM

Notes:

AG= Amber Glass

CERCLA- Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G= Glass

L = liter

MGP= Manufactured Gas Plant

ml = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

P= Poly

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition

USEPA or EPA= United States Environmental Protection Agency

VOA = volatile organic analysis

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

ZHS= Zero Head Space

^A For list of compounds in this analytical group, see series of Tables 1 and 2

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4A. Sampling and Analysis Summary - Test America
Water Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B	3 x 40 mL G-TLS, ZHS	Cool ≤6°C, HCl	1 x 40 mL	14 days
Diesel Range Organics	WI DRO	¹ 2 x 8oz AG WMG TLC	Cool ≤6°C, HCl	250 mL	7 days to extract 40 days to analyze
Gasoline Range Organics	WI GRO	3 x 40 mL G-TLS, ZHS	Cool ≤6°C, HCl	1 x 40 mL	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270D	¹ 2 x 8oz AG WMG TLC	Cool ≤6°C	250 mL	7 days to extract 40 days to analyze
Semivolatile Organic Compounds	SW846-8270D - low level	¹ 2 x 8oz AG WMG TLC	Cool ≤6°C	250 mL	7 days to extract 40 days to analyze
Semivolatile Organic Compounds	SW846-8270D SIM	2 x 1L-AG or WMG TLC	Cool ≤6°C	1L	7 days to extract 40 days to analyze
Semivolatile Organic Compounds	GC/MS Isotope Dilution - low level	2 x 1L-AG or WMG TLC	Cool ≤6°C	1L	7 days to extract 40 days to analyze
Phenols					
Phenols	SW846-8270C/D	¹ 2 x 8oz AG WMG TLC	Cool ≤6°C	250 mL	7 days to extract 40 days to analyze
Phenolics	SW846 9066	500 mL, AG, TLC	Cool ≤6°C, H ₂ SO ₄ pH < 2	50 mL	28 days
Pesticides					
Pesticides	SW846 8081B	¹ 2 x 8oz AG WMG TLC	Cool ≤6°C	250 mL	7 days to extract 40 days to analyze
PCBs					
PCBs	SW846 8082A	¹ 2 x 8oz AG WMG TLC	Cool ≤6°C	250 mL	365 days to extract 40 days to analyze
Inorganics					
Metals, total	SW846-6020A	250 mL, P	HNO ₃ to pH < 2	50 mL	180 days
Metals, dissolved	SW846-6020A	250 mL, P	HNO ₃ to pH < 2	50 mL	180 days
Chromium III	SM 3500_CR3_B	Calculation from Total Chromium - Chromium VI			
Chromium VI	SM 3500_CR3_B	500 mL, P	Cool ≤6°C	25 mL	24 hours unpreserved, 28 days preserved ²
Cyanide, total	SW846-9012A/9014	250 mL, P	Cool ≤6°C, NaOH pH > 12	50 mL	14 days
Cyanide, available	OIA-1677	Contact the TestAmerica laboratory performing the analysis for method-exclusive containers			14 days
Cyanide, amenable	SW846 9012B/9014	250 mL, P	Cool ≤6°C, NaOH pH > 12	50 mL	14 days
Cyanide, dissociable	SM 4500CN	250 mL, P	Cool ≤6°C, NaOH pH > 12	50 mL	14 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Mercury	SW846-7471B	250 mL, P	HNO ₃ to pH < 2	30 mL	28 days
Other					
Alkalinity as CaCO ₃	SM 2320B	1L, P (limited headspace)	Cool ≤6°C	100 mL	14 days
Ammonia	EPA Method 350.1	500 mL, P	Cool ≤6°C, H ₂ SO ₄	100 mL	28 days
Biochemical Oxygen Demand (BOD)	SM 5210B	1L, P	Cool ≤6°C	500 mL	48 hours
Chloride	SW846 9056A	250 mL, P	Cool ≤6°C	50 mL	28 days
Chemical Oxygen Demand (COD)	SM 5220C	500 mL, P	Cool ≤6°C, H ₂ SO ₄ to pH <2	100 mL	28 days
Fluoride	SM 4500F-C	1L, P	Cool <6°C	100 mL	28 days
Hardness	SW846 6010/SM 2340B Calc.	250 mL, P	HNO ₃ to pH < 2	50 mL	180 days
Methane	RSK-175	3 x 40 mL G-TLS, ZHS	³ Cool ≤6°C, HCl	1 x 40 mL	14 days
Nitrate	SW846 9056A	250 mL, P	Cool ≤6°C	50 mL	48 hours
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	500 mL, P	Cool ≤6°C, H ₂ SO ₄ to pH < 2	50 mL	28 days
Oil & Grease	EPA Method 1664A	2 x 1L-AG or WMG TLC	Cool ≤6°C, H ₂ SO ₄	1L	28 days
pH	EPA Method 150.1/ SM 9040/ SM 9045	1L P	Cool ≤6°C	50 mL	Immediate ⁴
Phosphate	SM 4500_P_E	500 mL, P	Cool ≤6°C, H ₂ SO ₄ pH < 2	100 mL	28 days
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	1L, P	Cool ≤6°C	1000 mL	7 days
Sulfate	SW846 9056A	250 mL, P	Cool ≤6°C	50 mL	28 days
Sulfur	SW846 6010	250 mL, P	HNO ₃ to pH < 2	50 mL	180 days
Pentachlorophenol	SW846-8151	2 x 1L-AG or WMG TLC	Cool ≤6°C	1L	7 days to extract 40 days to analyze
Total Organic Carbon (TOC)	SW846 9060	2 x 40 mL G-TLS, ZHS	Cool ≤6°C, H ₂ SO ₄ or HCl pH < 2	40 mL	28 days

O: SLM

C: SSW

F: SLM

Notes:

AG= Amber Glass

°C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G= Glass

GC= gas chromatograph

L = liter

MGP= Manufactured Gas Plant

mL = milliliter

MS= mass spectrometry

OIA= OI Corporation, Published in EPA/821-R-04-001

P= Poly

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP= standard operating procedure
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
TLC= Teflon-line cap
TLS= Teflon-lined septum
USEPA or EPA= United States Environmental Protection Agency
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
WMG= Wide-Mouth Glass
ZHS= Zero Head Space
< = less than
≤ = less than or equal to

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection.
^A For list of compounds in this analytical group, see series of Tables 1 and 2.
^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

¹ Large/High Volume Injection (LVI) Technique containers are as follows:
2 x 8-oz AG WMG TLC, or
2 x 4-oz AG WMG TLC or
2 x 40 mL AG TLS

² To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6
³ Sample temperature to be maintained at 0-6°C
⁴ Immediate equals 15 minutes from sampling or field test

Table 4A. Sampling and Analysis Summary - Eurofins
Water Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B	3 x 40 mL glass vials	HCl; 2-6C	120 mL	14 days
Diesel Range Organics	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C SIM	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
Semivolatile Organic Compounds	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
Phenols					
2,4-Dichlorophenol	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
2,4-Dimethylphenol	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
2-Methylphenol (o-cresol)	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
3&4-Methylphenol (m, p-cresol)	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
4-Methylphenol	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
Phenol	SW846-8270C/D	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
Phenolics	SW846 9066	250 mL glass	H ₂ SO ₄ ; 2-6C	100 mL	28 days
Pesticides					
Pesticides	SW846 8081B	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
PCBs					
PCBs	SW846 8082A	2 x 250 mL amber glass	2-6C	500 mL	7 days extraction/40 days analysis
Inorganics					
Metals, total	SW846-6020A/ 6010	250 mL plastic	HNO ₃ ; 2-6C	100 mL	6 months
Metals, dissolved	SW846-6020A/ 6010	250 mL plastic, field filtered	HNO ₃ ; 2-6C	100 mL	6 months
Chromium III	SW846-6020A/ 6011				
Chromium VI	SW846-6020A/ 6012				
Cyanide, total	SW846-9012A	250 mL plastic	NaOH/ascorbic acid; 2-6C	100 mL	14 days
Cyanide, available	OIA-1677	250 mL plastic	NaOH/ascorbic acid; 2-6C	100 mL	14 days
Cyanide, amenable	SW846 9012B/9014	250 mL plastic	NaOH/ascorbic acid; 2-6C	100 mL	14 days
Cyanide, dissociable	SM 4500CN	250 mL plastic	NaOH/ascorbic acid; 2-6C	100 mL	14 days
Mercury	SW846-7471B				
Other					
Alkalinity as CaCO ₃	SM 2320B	250 mL plastic	2-6C	120 mL	14 days
Ammonia	EPA Method 350.1	1000 mL plastic	H ₂ SO ₄ ; 2-6C	100 mL	28 days
Biochemical Oxygen Demand (BOD)	SM 5210B	1000 mL plastic	2-6C	250 mL	48 hours
Chloride	SM 4500-CI E	500 mL plastic	2-6C	200 mL	28 days
Chemical Oxygen Demand (COD)	EPA Method 410.4	1000 mL plastic	2-6C	250 mL	48 hours
Fluoride	SM 4500F-C	250 mL plastic	2-6C	120 mL	28 days
Methane	RSK-175	2 x 40 mL glass	HCl; 2-6C	80 mL	14 days
Nitrate	EPA Method 353.2	2 x 40 mL glass	1 - H ₂ SO ₄ ; 2-6C 1 - 2-6C	80 mL	48 hours extraction/ 28 days analysis
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	40 mL glass vial	H ₂ SO ₄ ; 2-6C	5 mL	28 days
Oil & Grease	EPA Method 1664A	2 x 1000 mL glass	HCl; 2-6C	1000 mL	28 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
pH	EPA Method 150.1/ SM 9040/ SM 9045	250 mL plastic	2-6C	120 mL	analyze immediately
Phosphate	EPA Method 365.1	250 mL plastic	H ₂ SO ₄ ; 2-6C	50 mL	28 days
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	1000 mL & 500 mL plastic	2-6C	1500 mL	7 days
Sulfate	SM 4500 SO ₄ E	250 mL plastic	2-6C	200 mL	28 days
Sulfur	SW846 6010	250 mL plastic	HNO ₃ ; 2-6C	100 mL	6 months
Pentachlorophenol	SW846-8151	2 x 1000 mL amber glass	2-6C	1000 mL	7 days extraction/40 days analysis
Total Organic Carbon (TOC)	SW846 9060	5 x 40 mL glass	H ₃ PO ₄ ; 2-6C	200 mL	28 days
Alternate Methods Provided					
PCBs	EPA Method 1668A/C	2 x 1000 mL amber glass	2-6C	2000 mL	365 days
Chromium VI	SW846 7196A	250 mL plastic	2-6C	150 mL	24 hours
Mercury	SW846 7470A	250 mL plastic	HNO ₃ ; 2-6C	100 mL	28 days
pH	EPA 150.1, SW 9040, SM 4500 H, B	250 mL plastic	2-6C	120 mL	analyze immediately
Hardness	SM 2340C	250 mL plastic	HNO ₃	100 mL	6 months
Sulfate	EPA 375.4	250 mL plastic	2-6C	100 mL	28 days

O: SLM

C: SSW

F: SLM

Notes:

C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

L = liter

MGP= Manufactured Gas Plant

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4A. Sampling and Analysis Summary - Pace Analytical
Water Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
Diesel Range Organics	WI DRO	1-1000mL Amber Glass	HCL	1-950mL	7 days
Gasoline Range Organics	WI GRO	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270 SIM	2-100mL Amber WMG	None ^C	2-100mL	7 days
Semivolatile Organic Compounds	SW846-8270 C/D	1-1000mL Amber Glass	None ^C	1-1000mL	7 days
Semivolatile Organic Compounds	Alkylated, SW846-8270 MOD	1-1000mL Amber Glass	None ^C	1-1000mL	7 days
Phenols					
Phenols	SW846-8270C/D	1-1000mL Amber Glass	None ^C	1-1000mL	7 days
Phenolics	SW846 9066				
Pesticides					
Pesticides	SW846 8081B	1-1000mL Amber Glass	None ^C	1-1000mL	7 days
PCBs					
PCBs	SW846 8082A	1-1000mL Amber Glass	None ^C	1-1000mL	365 days
Inorganics					
Metals, total	SW846-6020A/ 6010	1-250mL Plastic	HNO3	50mL	6 months
Metals, dissolved	SW846-6020A/ 6010	1-250mL Plastic	HNO3	50mL	6 months
Chromium III	SW846-6020A/ 6011				
Chromium VI	SW846-6020A/ 6012	1-250mL Plastic	None ^C	40mL	24 hours
Cyanide, total	SW846-9012A	1-250mL Plastic	NaOH	10mL	14 days
Cyanide, available	OIA-1677	1-250mL Plastic	NaOH	100mL	14 days
Cyanide, amenable	SW846 9012B/9014	1-250mL Plastic	NaOH	100mL	14 days
Cyanide, dissociable	SM 4500CN	1-250mL Plastic	NaOH	60mL	14 Days
Mercury	SW846-7471B	1-250mL Plastic	HNO3	10mL	28 Days
Other					
Alkalinity as CaCO ₃	SM 2320B	1-250mL Plastic	None ^C	20mL	14 days
Alkalinity as CaCO ₃	EPA Method 310.2	1-250mL Plastic	None ^C	20mL	14 days
Alkalinity as CaCO ₃	SM 5310C				
Ammonia	EPA Method 350.1	1-250mL Plastic	H2SO4	10mL	28 days
Biochemical Oxygen Demand (BOD)	SM 5210B	1-1000mL Plastic	None ^C	600mL	48 hours
Chloride	SM 4500-Cl E				
Chemical Oxygen Demand (COD)	EPA Method 410.4	1-250mL Plastic	H2SO4	10mL	28 days
Fluoride	SM 4500F-C				
Methane	RSK-175	3-40mL VOA Vials	None ^C	40mL	7 Days
Nitrate	EPA Method 353.2	1-250mL Plastic	H2SO4	10mL	28 days
Nitrogen, Nitrate & Nitrite	EPA Method 353.2	1-250mL Plastic	H2SO4	10mL	28 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Oil & Grease	EPA Method 1664A	1-1000mL Amber Glass	HCL/H2SO4	1-1000mL	28 days
pH	EPA Method 150.1/ SM 9040/ SM 9045	1-250mL Plastic	None ^C	70mL	15 minutes
Phosphate	EPA Method 365.1/365.4	1-250mL Plastic	H2SO4	20mL	28 days
Residue, Non-filterable Total Suspended Solids (TSS)	SM 2540D	1-1000mL Plastic	None ^C	500mL	7 days
Sulfate	SM 4500 SO4 E				
Pentachlorophenol	SW846-8151	1-1000mL Amber Glass	None ^C	1000mL	7 days
Total Organic Carbon (TOC)	SW846 9060	1-125mL Amber Glass	H2SO4	60mL	28 days
Total Organic Carbon (TOC)	EPA Method 310.2				
Total Organic Carbon (TOC)	SM 5310C	1-125mL Amber Glass	H2SO4	60mL	28 days
Alternate Methods Provided					
Phenolics	EPA Method 420.4	1-125ml Amber Glass	H2SO4	70mL	28 days
Chrome VI	SM 3500Cr-D	1-250mL Plastic	None ^C	40mL	24 hours
Mercury	EPA Method 7470A	1-250mL Plastic	HNO3	10mL	28 days
Chloride	EPA Method 300.0 / SM 9056	1-250mL Plastic	None ^C	10mL	28 days
Fluoride	EPA Method 300.0 / SM 9056	1-250mL Plastic	None ^C	10mL	28 days
Methane	SW846-8015B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
Nitrate	EPA Method 300.0 / SM 9056	1-250mL Plastic	None ^C	10mL	28 days
Sulfate	EPA Method 300.0 / SM 9056	1-250mL Plastic	None ^C	10mL	28 days
1,2-Dichloroethene	SW846-8260B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
cis-1,2-Dichloroethene	SW846-8260B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
trans-1,2-Dichloroethene	SW846-8260B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
p-Isopropyltoluene	SW846-8260B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
Dichloromethane	SW846-8260B	3-40mL VOA Vials	HCL	1-40mL VOA Vial	14 days
Phenolics	EPA Method 420.4	1-250mL Amber Glass	H2SO4	50mL	28 days

O: SLM

C: SSW

F: SLM

Notes:

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

MGP= Manufactured Gas Plant

mL = milliliter

MOD= modified method

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

VOA = volatile organic analysis

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4B. Sampling and Analysis Summary - Alpha
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B	3-40 mL G TLS	2-Water (freeze)/1-MeOH	5 g (1:1 with preservative)	14 days
Diesel Range Organics	WI DRO	1-120 mL G TLC	none ^C	100 g	14 days
Gasoline Range Organics	WI GRO	1-40 mL G TLS	MeOH	5 g (1:1 with preservative)	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	1-120 mL G TLC	none ^C	100 g	14 days
Phenols					
Phenols	SW846-8270C	1-120 mL G TLC	none ^C	100 g	14 days
Pesticides					
Pesticides	SW846-8081	1-250 mL G TLC	none ^C	100 g	14 days
PCBs					
PCBs	SW846-8082	1-250 mL G TLC	none ^C	100 g	14 days
Indicator Parameters					
Soot Carbon	none	1-250 mL G TLC	none ^C	100 g	28 days
Total Organic Carbon (TOC)	SW846 9060A	1-250 mL G TLC	none ^C	100 g	28 days
Fraction Organic Carbon (FOC)	ASTM D2974	1-250 mL G TLC	none ^C	100 g	28 days
Inorganics					
Metals, total	SW846-6020A	1-120 mL G TLC	none ^C	100 g	180 days
Cyanide, total	SW846-9012A	1-250 mL G TLC	none ^C	200 g	14 days
Cyanide, available	OIA-1677	1-250 mL G TLC	none ^C	200 g	14 days
Iron	SW846-6020A	1-120 mL G TLC	none ^C	100 g	180 days
Lead	SW846-6020A	1-120 mL G TLC	none ^C	100 g	180 days
Manganese	SW846-6020A	1-120 mL G TLC	none ^C	100 g	180 days
Mercury	SW846-7471A/B	1-250 mL G TLC	none ^C	200 g	28 days
Other					
Black carbon (Gustafsson)	Gustafsson Method	1-250 mL G TLC	none ^C	200 g	14 days
Alkalinity	SM 2320B				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Ammonia	EPA Method 350.1	1-250 mL G TLC	none ^C	200 g	28 days
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937	1-250 mL G TLC	none ^C	200 g	N/A
Chloride	EPA Method 325.2	1-250 mL G TLC	none ^C	200 g	28 days
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Cyanide, Reactive	SW846 7.3.3.2	1-250 mL G TLC	none ^C	200 g	14 days
Flash Point	EPA Method 1010/1020	1-250 mL G TLC	none ^C	200 g	
Fluoride	SM 4500F-C	1-250 mL G TLC	none ^C	200 g	14 days
Free Liquids (Paint Filter)	SW846 9095B	1-250 mL G TLC	none ^C	200 g	N/A
Moisture Content	SW846 3550C	1-250 mL G TLC	none ^C	200 g	N/A
Nitrate/Nitrite	EPA Method 353.2	1-250 mL G TLC	none ^C	200 g	28 days
Total Kjeldahl Nitrogen	SM 4500-NH3 G	1-250 mL G TLC	none ^C	200 g	28 days
pH	SW846 9045D	1-250 mL G TLC	none ^C	200 g	24 hours
Phenolics	SW846 9066				
Phosphate	EPA Method 365.1	1-250 mL G TLC	none ^C	200 g	28 days
Total Phosphorus	EPA Method 365.1	1-250 mL G TLC	none ^C	200 g	28 days
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E	1-250 mL G TLC	none ^C	200 g	
Sulfur	SW846 6010	1-120 mL G TLC	none ^C	100 g	180 days
Sulfur, Reactive	SW846 7.3.4.2				
Total Organic Carbon (TOC)	ASTM-2974-00	1-250 mL G TLC	none ^C	200 g	28 days
Total Organic Carbon (TOC)	Lloyd Kahn Method	1-250 mL G TLC	none ^C	200 g	14 days
Total Organic Carbon (TOC)	Walkley Black Method				
Total Organic Carbon (TOC)	SW846-9060M	1-250 mL G TLC	none ^C	200 g	28 days
Oil and Grease	SW846-9071B	1-250 mL G TLC	none ^C	200 g	28 days
Pentachlorophenol	SW846-8151	1-250 mL G TLC	none ^C	200 g	14 days
Alternate Methods Provided					
Ammonia	SM 4500NH3	1-250 mL G TLC	none ^C	200 g	28 days
Moisture Content	SM 2540G	1-250 mL G TLC	none ^C	200 g	N/A
Nitrate/Nitrite	SM 4500NO3-F	1-250 mL G TLC	none ^C	200 g	28 days
Phosphate	SM 4500P-E(M)	1-250 mL G TLC	none ^C	200 g	28 days
Total Phosphorus	SM 4500P-E	1-250 mL G TLC	none ^C	200 g	28 days

O: SLM

C: SSW

F: SLM

Notes:

ASTM = ASTM International

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G= Glass

g = gram

MGP= Manufactured Gas Plants

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition.

TLC= Teflon-lined cap

TLS= Teflon-lined septum

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2.

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection.

Table 4B. Sampling and Analysis Summary - Brighton
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B	9 oz. Glass Jar-Methanol Preservation Kit	Methanol	10ml Methanol	14 days
Diesel Range Organics	WI DRO	9 oz. Glass Jar	None ^C	9 oz.	14 days
Gasoline Range Organics	WI GRO	9 oz. Glass Jar-Methanol Preservation Kit	Methanol	10ml Methanol	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	9 oz. Glass Jar	None ^C	9 oz.	Extract within 14 days/Analyze within 40 days of extraction
Phenols					
Phenols	SW846-8270C	9 oz. Glass Jar	None ^C	9 oz.	Extract within 14 days/Analyze within 40 days of extraction
Pesticides					
Pesticides	SW846-8081	9 oz. Glass Jar	None ^C	9 oz.	Extract within 14 days/Analyze within 40 days of extraction
PCBs					
PCBs	SW846-8082	9 oz. Glass Jar	None ^C	9 oz.	Extract within 14 days/Analyze within 40 days of extraction
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846 9060A				
Fraction Organic Carbon (FOC)	ASTM D2974				
Inorganics					
Metals, total	SW846-6020A	9 oz. Glass Jar	None ^C	9 oz.	Analyze within 6 months
Cyanide, total	SW846-9012A	9 oz. Glass Jar	None ^C	9 oz.	28 days
Cyanide, available	OIA-1677	9 oz. Glass Jar	None ^C	9 oz.	28 days
Iron	SW846-6020A	9 oz. Glass Jar	None ^C	9 oz.	6 months
Lead	SW846-6020A	9 oz. Glass Jar	None ^C	9 oz.	6 months
Manganese	SW846-6020A	9 oz. Glass Jar	None ^C	9 oz.	6 months
Mercury	SW846-7471A/B	9 oz. Glass Jar	None ^C	9 oz.	6 months
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B	9 oz. Glass Jar	None ^C	9 oz.	14 days
Ammonia	EPA Method 350.1	9 oz. Glass Jar	None ^C	9 oz.	14 days
Biological Oxygen Demand (BOD)	SM 5210B	9 oz. Glass Jar	None ^C	9 oz.	48 hours
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937				
Chloride	EPA Method 325.2	9 oz. Glass Jar	None ^C	9 oz.	28 days
Chemical Oxygen Demand (COD)	EPA Method 410.4	9 oz. Glass Jar	None ^C	9 oz.	28 days
Cyanide, Reactive	SW846 7.3.3.2	9 oz. Glass Jar	None ^C	9 oz.	28 days
Flash Point	EPA Method 1010/1020	9 oz. Glass Jar	None ^C	9 oz.	28 days
Fluoride	SM 4500F-C	9 oz. Glass Jar	None ^C	9 oz.	28 days
Free Liquids (Paint Filter)	SW846 9095B	9 oz. Glass Jar	None ^C	9 oz.	28 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Moisture Content	SW846 3550C	9 oz. Glass Jar	None ^C	9 oz.	28 days
Nitrate/Nitrite	EPA Method 353.2	9 oz. Glass Jar	None ^C	9 oz.	28 days
Total Kjeldahl Nitrogen	SM 4500-NH3 G	9 oz. Glass Jar	None ^C	9 oz.	28 days
pH	SW846 9045D	9 oz. Glass Jar	None ^C	9 oz.	28 days
Phenolics	SW846 9066	9 oz. Glass Jar	None ^C	9 oz.	28 days
Phosphate	EPA Method 365.1	9 oz. Glass Jar	None ^C	9 oz.	28 days
Total Phosphorus	EPA Method 365.1	9 oz. Glass Jar	None ^C	9 oz.	28 days
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E	9 oz. Glass Jar	None ^C	9 oz.	28 days
Sulfur	SW846 6010				
Sulfur, Reactive	SW846 7.3.4.2				
Total Organic Carbon (TOC)	ASTM-2974-00				
Total Organic Carbon (TOC)	Lloyd Kahn Method				
Total Organic Carbon (TOC)	Walkley Black Method				
Total Organic Carbon (TOC)	SW846-9060M				
Oil and Grease	SW846-9071B				
Pentachlorophenol	SW846-8151	9 oz. Glass Jar	None ^C	9 oz.	14 days
Alternate Methods Provided					
Ammonia	SM 4500NH3	9 oz. Glass Jar	None ^C	9 oz.	28 days
Moisture Content	SM 2540G	9 oz. Glass Jar	None ^C	9 oz.	28 days
Nitrate/Nitrite	SM 4500NO3-F	9 oz. Glass Jar	None ^C	9 oz.	28 days
Phosphate	SM 4500P-E(M)	9 oz. Glass Jar	None ^C	9 oz.	28 days
Total Phosphorus	SM 4500P-E	9 oz. Glass Jar	None ^C	9 oz.	28 days
			O: SLM	C: SSW	F: SLM

Notes:

ASTM - ASTM International

CERCLA= Comprehensive Environmental Response, Compensation, and Reliability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

MGP= Manufactured Gas Plant

ml = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

oz = ounce

PAH = polycyclic aromatic

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4B. Sampling and Analysis Summary - STAT
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B	5350 Preserved Kit	Methanol/Sodium bisulfate	5 grams per Vial	14 Days
Diesel Range Organics	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	4oz Glass jar	Cool 4°C	50 grams	14 Days
Phenols					
Phenols	SW846-8270C	4oz Glass jar	Cool 4°C	50 grams	14 Days
Pesticides					
Pesticides	SW846-8081	4oz Glass jar	Cool 4°C	50 grams	14 Days
PCBs					
PCBs	SW846-8082	4oz Glass jar	Cool 4°C	50 grams	None
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846 9060A				
Fraction Organic Carbon (FOC)	ASTM D2974	4oz Glass jar	None ^C	50 grams	None
Inorganics					
Metals, total	SW846-6020A	4oz Glass jar	Cool 4°C	50 grams	180 Days
Cyanide, total	SW846-9012A	4oz Glass jar	Cool 4°C	50 grams	14 Days
Cyanide, available	OIA-1677				
Iron	SW846-6020A	4oz Glass jar	Cool 4°C	50 grams	180 Days
Lead	SW846-6020A	4oz Glass jar	Cool 4°C	50 grams	180 Days
Manganese	SW846-6020A	4oz Glass jar	Cool 4°C	50 grams	180 Days
Mercury	SW846-7471A/B	4oz Glass jar	Cool 4°C	50 grams	28 Days
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B	4oz Glass jar	Cool 4°C	50 grams	14 Days
Ammonia	EPA Method 350.1	4oz Glass jar	Cool 4°C	50 grams	28 Days
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937	16oz Glass Jar	None ^C	250 grams	None
Chloride	EPA Method 325.2	4oz Glass jar	Cool 4°C	50 grams	28 Days
Chemical Oxygen Demand (COD)	EPA Method 410.4				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Cyanide, Reactive	SW846 7.3.3.2	4oz Glass jar	Cool 4°C	50 grams	14 Days
Flash Point	EPA Method 1010/1020	4oz Glass jar	Cool 4°C	50 grams	30 Days
Fluoride	SM 4500F-C	4oz Glass jar	Cool 4°C	50 grams	28 Days
Free Liquids (Paint Filter)	SW846 9095B	4oz Glass jar	Cool 4°C	50 grams	None
Moisture Content	SW846 3550C	4oz Glass jar	None ^C	50 grams	None
Nitrate/Nitrite	EPA Method 353.2	4oz Glass jar	Cool 4°C	50 grams	2 Days
Total Kjeldahl Nitrogen	SM 4500-NH3 G				
pH	SW846 9045D	4oz Glass jar	Cool 4°C	50 grams	14 Days
Phenolics	SW846 9066	4oz Glass jar	Cool 4°C	50 grams	28 Days
Phosphate	EPA Method 365.1	4oz Glass jar	Cool 4°C	50 grams	28 Days
Total Phosphorus	EPA Method 365.1	4oz Glass jar	Cool 4°C	50 grams	28 Days
Specific Gravity	ASTM D5057	4oz Glass jar	None ^C	50 grams	None
Sulfate	SM 4500-SO4 E	4oz Glass jar	Cool 4°C	50 grams	28 Days
Sulfur	SW846 6010				
Sulfur, Reactive	SW846 7.3.4.2	4oz Glass jar	Cool 4°C	50 grams	7 Days
Total Organic Carbon (TOC)	ASTM-2974-00	4oz Glass jar	None ^C	50 grams	None
Total Organic Carbon (TOC)	Lloyd Kahn Method				
Total Organic Carbon (TOC)	Wakley Black Method				
Total Organic Carbon (TOC)	SW846-9060M				
Oil and Grease	SW846-9071B	4oz Glass jar	Cool 4°C	50 grams	28 Days
Pentachlorophenol	SW846-8151				
Alternate Methods Provided					
Ammonia	SM 4500NH3	4oz Glass jar	Cool 4°C	50 grams	28 Days
Moisture Content	SM 2540G	4oz Glass jar	Cool 4°C	50 grams	7 Days
Nitrate/Nitrite	SM 4500NO3-F	4oz Glass jar	Cool 4°C	50 grams	28 Days
Phosphate	SM 4500P-E(M)	4oz Glass jar	Cool 4°C	50 grams	28 Days
Total Phosphorus	SM 4500P-E	4oz Glass jar	Cool 4°C	50 grams	28 Days

O: SLM

C: SSW

F: SLM

Notes:

ASTM - ASTM International

°C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

OIA= OI Corporation, Published in EPA/821-R-04-001

MGP= Manufactured Gas Plant

oz = ounce

PAH = polycyclic aromatic

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition.

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2.

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection.

Table 4B. Sampling and Analysis Summary - Battelle
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B				
Diesel Range Organics	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	G with TLC	Cool <6C or freeze <-10C	100g	Cool - 14 days; Frozen - 1 year
Phenols					
Phenols	SW846-8270C				
Pesticides					
Pesticides	SW846-8081				
PCBs					
PCBs	SW846-8082				
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846 9060A				
Fraction Organic Carbon (FOC)	ASTM D2974				
Inorganics					
Metals, total	SW846-6020A				
Cyanide, total	SW846-9012A				
Cyanide, available	OIA-1677				
Iron	SW846-6020A				
Lead	SW846-6020A				
Manganese	SW846-6020A				
Mercury	SW846-7471A/B				
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B				
Ammonia	EPA Method 350.1				
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937				
Chloride	EPA Method 325.2				
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Cyanide, Reactive	SW846 7.3.3.2				
Flash Point	EPA Method 1010/1020				
Fluoride	SM 4500F-C				
Free Liquids (Paint Filter)	SW846 9095B				
Moisture Content	SW846 3550C				
Nitrate/Nitrite	EPA Method 353.2				
Total Kjeldahl Nitrogen	SM 4500-NH3 G				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
pH	SW846 9045D				
Phenolics	SW846 9066				
Phosphate	EPA Method 365.1				
Total Phosphorus	EPA Method 365.1				
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E				
Sulfur	SW846 6010				
Sulfur, Reactive	SW846 7.3.4.2				
Total Organic Carbon (TOC)	ASTM-2974-00				
Total Organic Carbon (TOC)	Lloyd Kahn Method				
Total Organic Carbon (TOC)	Walkley Black Method				
Total Organic Carbon (TOC)	SW846-9060M				
Oil and Grease	SW846-9071B				
Pentachlorophenol	SW846-8151				
Alternate Methods Provided					
Ammonia	SM 4500NH3				
Moisture Content	SM 2540G				
Nitrate/Nitrite	SM 4500NO3-F				
Phosphate	SM 4500P-E(M)				
Total Phosphorus	SM 4500P-E				

O: SLM

C: SSW

F: SLM

Notes:

ASTM - ASTM International

C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G= Glass

g = gram

MGP= Manufactured Gas Plant

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

TLC= Teflon-lined cap

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

< = less than

^A For list of compounds in this analytical group, see series of Tables 1 and 2.^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4B. Sampling and Analysis Summary - ESS
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B	2x40mL VOA	MeOH/Freeze	2x40mL VOA	14 days
Diesel Range Organics	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	8oz AG	None ^C	4oz AG	14 days
Phenols					
Phenols	SW846-8270C	8oz AG	None ^C	4oz AG	14 days
Pesticides					
Pesticides	SW846-8081	8oz AG	None ^C	4oz AG	14 days
PCBs					
PCBs	SW846-8082	8oz AG	None ^C	4oz AG	14 days
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846 9060A	4oz AG	None ^C	2oz AG	28 days
Fraction Organic Carbon (FOC)	ASTM D2974				
Inorganics					
Metals, total	SW846-6020A	4oz AG	None ^C	2oz AG	6 months
Cyanide, total	SW846-9012A	4oz AG	None ^C	2oz AG	14 days
Cyanide, available	OIA-1677	4oz AG	None ^C	2oz AG	14 days
Iron	SW846-6020A	4oz AG	None ^C	2oz AG	6 months
Lead	SW846-6020A	4oz AG	None ^C	2oz AG	6 months
Manganese	SW846-6020A	4oz AG	None ^C	2oz AG	6 months
Mercury	SW846-7471A/B	4oz AG	None ^C	2oz AG	28 days
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B				
Ammonia	EPA Method 350.1				
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Chloride	EPA Method 325.2				
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Cyanide, Reactive	SW846 7.3.3.2	4oz AG	None ^C	2oz AG	14 days
Fluoride	SM 4500F-C				
Free Liquids (Paint Filter)	SW846 9095B	4oz AG	None ^C	4oz AG	28 days
Moisture Content	SW846 3550C	4oz AG	None ^C	2oz AG	14 days
Nitrate/Nitrite	EPA Method 353.2				
Total Kjeldahl Nitrogen	SM 4500-NH3 G				
pH	SW846 9045D	4oz AG	None ^C	2oz AG	Immediate
Phenolics	SW846 9066				
Phosphate	EPA Method 365.1				
Total Phosphorus	EPA Method 365.1				
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E				
Sulfur	SW846 6010				
Sulfur, Reactive	SW846 7.3.4.2	4oz AG	None ^C	2oz AG	14 days
Total Organic Carbon (TOC)	ASTM-2974-00	4oz AG	None ^C	2oz AG	14 days
Total Organic Carbon (TOC)	Lloyd Kahn Method	4oz AG	None ^C	2oz AG	28 days
Total Organic Carbon (TOC)	Walkley Black Method				
Total Organic Carbon (TOC)	SW846-9060M	4oz AG	None ^C	2oz AG	28 days
Oil and Grease	SW846-9071B	4oz AG	None ^C	4oz AG	14 days
Pentachlorophenol	SW846-8151				
Alternate Methods Provided					
Ammonia	4500NH3				
Moisture Content	2540G				
Nitrate/Nitrite	4500NO3-F				
Phosphate	4500P-E(M)				
Total Phosphorus	4500P-E				

O: SLM

C: SSW

F: SLM

Notes:

AG= Amber Glass

ASTM - ASTM International

°C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

MGP= Manufactured Gas Plant

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

oz = ounce

PAH = polycyclic aromatic

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition.

USEPA or EPA= United States Environmental Protection Agency

VOA = volatile organic analysis

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

^A For list of compounds in this analytical group, see series of Tables 1 and 2.

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

^C Sample should be cooled to a temperature range of 2 to 6 degrees Celsius, and shipped on ice

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection.

Table 4B. Sampling and Analysis Summary - EERC

Soil/Sediment Matrix

MGP Multi-Site Program

USEPA Region 5

CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B/ 8021B				
Diesel Range Organics	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	AG, TLS	Cool to 4 C	250 mL	28 days to prep
Phenols					
Phenols	SW846-8270C				
Pesticides					
Pesticides	SW846-8081				
PCBs					
PCBs	SW846-8082				
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846 9060A				
Fraction Organic Carbon (FOC)	ASTM D2974				
Inorganics					
Metals, total	SW846-6020A				
Cyanide, total	SW846-9012A				
Cyanide, available	OIA-1677				
Iron	SW846-6020A				
Lead	SW846-6020A				
Manganese	SW846-6020A				
Mercury	SW846-7471A/B				
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B				
Ammonia	EPA Method 350.1				
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937				
Chloride	EPA Method 325.2				
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Cyanide, Reactive	SW846 7.3.3.2				
Flash Point	EPA Method 1010/1020				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Fluoride	SM 4500F-C				
Free Liquids (Paint Filter)	SW846 9095B				
Moisture Content	SW846 3550C				
Nitrate/Nitrite	EPA Method 353.2				
Total Kjeldahl Nitrogen	SM 4500-NH3 G				
pH	SW846 9045D				
Phenolics	SW846 9066				
Phosphate	EPA Method 365.1				
Total Phosphorus	EPA Method 365.1				
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E				
Sulfur	SW846 6010				
Sulfur, Reactive	SW846 7.3.4.2				
Total Organic Carbon (TOC)	ASTM-2974-00				
Total Organic Carbon (TOC)	Lloyd Kahn Method				
Total Organic Carbon (TOC)	Walkley Black Method				
Total Organic Carbon (TOC)	SW846-9060M				
Oil and Grease	SW846-9071B				
Pentachlorophenol	SW846-8151				
Alternate Methods Provided					
Ammonia	SM 4500NH3				
Moisture Content	SM 2540G				
Nitrate/Nitrite	SM 4500NO3-F				
Phosphate	SM 4500P-E(M)				
Total Phosphorus	SM 4500P-E				

O: SLM

C: SSW

F: SLM

Notes:

AG= Amber Glass

ASTM - ASTM International

C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

MGP= Manufactured Gas Plant

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

PAH = polycyclic aromatic

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

TLS= Teflon-lined septum

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

< = less than

^A For list of compounds in this analytical group, see series of Tables 1 and 2

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4B. Sampling and Analysis Summary - Test America
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Petroleum Volatile Organic					
Petroleum Volatile Organic Compounds	SW846-8260B	2-oz. jar; Full TerraCore Kit ¹	Cool <6°C, MeOH/ Na ₂ S ₂ O ₃ / H ₂ O; Frozen w/in 48 hours	10 g	14 days
Diesel Range Organics	WI DRO	4-oz. jar (certified tare wt.)	Cool ≤6°C	30 g	14 days to extract 40 days to analyze
Gasoline Range Organics	WI GRO	1 x 40 mL G-TLS w/10mL MeOH, syringe	Cool ≤6°C	1 x 40 mL	21 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270D	8-oz. jar	Cool ≤6°C	30 g	14 days to extract 40 days to analyze
Semivolatile Organic Compounds	SW846-8270D low level	8-oz. jar	Cool ≤6°C	30 g	14 days to extract 40 days to analyze
Semivolatile Organic Compounds	SW846-8270D SIM	8-oz. jar, Amber Wide	Cool ≤6°C	60 g	14 days to extract 40 days to analyze
Phenols					
Phenols	SW846-8270C	8-oz. jar	Cool ≤6°C	30 g	14 days to extract 40 days to analyze
Pesticides					
Pesticides	SW846-8081A/B	8-oz. jar	Cool ≤6°C	30 g	14 days to extract 40 days to analyze
PCBs					
PCBs	SW846-8082A	8-oz. jar	Cool ≤6°C	30 g	365 days to extract 40 days to analyze
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846-9060A/ASTM D4129- 82M/ASTM D2974/Lloyd Kahn Method	8-oz. jar/ 8-oz. jar, Amber (Lloyd Kahn)	Cool ≤6°C	10g	28 days 14 days (Lloyd Kahn)
Fractional Organic Carbon (FOC)	SW846-9060A/ASTM D4129- 82M/ASTM D2974/Lloyd Kahn Method	8-oz. jar	Cool ≤6°C	10 g	28 days
Inorganics					
Metals	SW846-6010B	8-oz. jar	Cool ≤6°C	5 g	180 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Metals	SW846-6020A	8-oz. jar	Cool ≤6°C	5 g	180 days
Cyanide, total	SW846-9012A/9014	8-oz. jar	Cool ≤6°C	5 g	14 days
Cyanide, available	OIA-1677	Contact the TestAmerica laboratory performing the analysis for method-exclusive containers			14 days
Mercury	SW846-7471A/B	8-oz. jar	Cool ≤6°C	10 g	28 days
Other					
Black carbon	Lloyd Kahn Method	8-oz. jar	Cool ≤6°C	10g	none
Alkalinity	SM 2320B	8-oz. jar	Cool ≤6°C	25 g	14 days ²
Ammonia	EPA Method 350.1	8-oz. jar	Cool ≤6°C	100 g	28 days ²
Biological Oxygen Demand (BOD)	SM 5210B	16 oz. jar	Cool ≤6°C	150 g	48 hours ²
British Therman Unit (BTU)	ASTM D240	8-oz. jar	Cool ≤6°C	50 g	none
Bulk Density	SM2710F	8-oz. jar	Cool ≤6°C	30g	28 days
Chloride	SW846-9056A	8-oz. jar	Cool ≤6°C	50 g	28 days ²
Chemical Oxygen Demand (COD)	SM5220C	8-oz. jar	Cool ≤6°C	25 g	28 days ²
Cyanide, Total	SW846-9010B/9014	8-oz. jar	Cool ≤6°C	5 g	14 days
Flash Point	EPA Method 1010/1020	8-oz. jar	Cool ≤6°C	150 g	none
Fluoride	SM 4500F-C	8-oz. jar	Cool ≤6°C	25 g	28 days ²
Free Liquids (Paint Filter)	SW846-9095B	8-oz. jar	Cool ≤6°C	125 g	none
Moisture Content	SM2540G	8-oz. jar	Cool ≤6°C	20 g	14 days
Nitrate/Nitrite	EPA Method 353.2	8-oz. jar	Cool ≤6°C	25 g	28 days ²
Total Kjeldahl Nitrogen	SM 4500-NH3 G	8-oz. jar	Cool ≤6°C	25 g	28 days ²
pH	SW846-9045D	8-oz. jar	Cool ≤6°C	30g	IMMEDIATELY ³
Phenolics	SW846-9066	8-oz. jar	Cool ≤6°C	5 g	28 days
Phosphorus as Phosphate	SM4500_P_E	8-oz. jar	Cool ≤6°C	5 g	28 days
Total Phosphorus	SM4500_P_E	8-oz. jar	Cool ≤6°C	5 g	28 days
Specific Gravity	SM2710F	8-oz. jar	Cool ≤6°C	30g	28 days
Sulfate	SW846-9056A	8-oz. jar	Cool ≤6°C	50 g	28 days ²
Sulfur	SW846-6010	8-oz. jar	Cool ≤6°C	5 g	180 days
Sulfide	SW846 9034/9030	8-oz. jar	Cool ≤6°C	50 g	7 days
Total Organic Carbon (TOC)	Lloyd Kahn Method	8-oz. jar	Cool ≤6°C	10 g	14 days
Total Organic Carbon (TOC)	Walkley Black Method	8-oz. jar	Cool ≤6°C	50 g	28 days
Total Organic Carbon (TOC)	SW846-9060M	8-oz. jar	Cool ≤6°C	10 g	28 days
Oil and Grease	SW846-9071B	8-oz. jar	Cool ≤6°C	30g	28 days
Pentachlorophenol	SW846-8151	8-oz. jar	Cool ≤6°C	30 g	14 days to extract 40 days to analyze

O: SLM

C: SSW

F: SLM

Notes:

ASTM - ASTM International

°C = degrees Celsius

CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136

G= Glass

g = gram

MGP= Manufactured Gas Plant

mL = milliliter

OIA= OI Corporation, Published in EPA/821-R-04-001

oz = ounce

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SOP= standard operating procedure

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition

TLS= Teflon-lined septum

USEPA or EPA= United States Environmental Protection Agency

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

< = less than

≤ = less than or equal to

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

^A For list of compounds in this analytical group, see series of Tables 1 and 2.

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

¹ Additional soil volume of 10-20 grams is required for Total Solids determination. This can be collected in a 2 oz.glass jar or a 60 mL plastic bottle.

² For a Solid/Waste matrix, some inorganic parameters will undergo a DI water leach prior to analysis

³ Immediate equals 15 minutes from sampling or field test

Table 4B. Sampling and Analysis Summary - Eurofins
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Petroleum Volatile Organic					
Petroleum Volatile Organic Compounds	SW846-8260B/ 8021B	3 x 40 mL glass vials	Methanol, DI water, or Sodium Bisulfate; 2-6C	15 g for 8260; 5 g for 8021	14 days
Diesel Range Organics	WI DRO				
Gasoline Range Organics	WI GRO				
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846-8270C/D/SIM PAH	4 or 8 oz glass jar	2-6C	100 g	14 days extraction/40 days analysis
Phenols					
Phenols	SW846-8270C	4 or 8 oz glass jar	2-6C	100 g	14 days extraction/40 days analysis
Pesticides					
Pesticides	SW846 8081A	4 or 8 oz glass jar	2-6C	100 g	14 days extraction/40 days analysis
Pesticides	SW846 8081B	4 or 8 oz glass jar	2-6C	100 g	14 days extraction/40 days analysis
PCBs					
PCBs	SW846-8082A	4 or 8 oz glass jar	2-6C	100 g	14 days extraction/40 days analysis
Indicator Parameters					
Soot Carbon	none				
Total Organic Carbon (TOC)	SW846 9060A	4 or 8 oz glass jar	2-6C	20 g	28 days
Fraction Organic Carbon (FOC)	ASTM D2974				
Inorganics					
Metals	SW846-6020A	4 or 8 oz glass jar	2-6C	5 g	6 months
Metals	SW846-6010B	4 or 8 oz glass jar	2-6C	5 g	6 months
Metals	SW846-6010C/D	4 or 8 oz glass jar	2-6C	5 g	6 months
Cyanide, total	SW846-9012A	4 or 8 oz glass jar	2-6C	5 g	14 days
Cyanide, available	OIA-1677				
Mercury	SW846-7471A/B	4 or 8 oz glass jar	2-6C	5 g	28 days
Nickel	SW846-6020A	4 or 8 oz glass jar	2-6C	5 g	6 months
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B				

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Ammonia	EPA Method 350.1	4 or 8 oz glass jar	2-6C	100 g	28 days
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937				
Chloride	EPA Method 325.2				
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Cyanide, Reactive	SW846 7.3.3.2	4 oz glass jar	2-6C	100 g	none
Flash Point	EPA Method 1010/1020	4 or 8 oz glass jar	2-6C	250 g	30 days
Fluoride	SM 4500F-C	4 or 8 oz glass jar	2-6C	50 g	28 days
Free Liquids (Paint Filter)	SW846 9095B	4 or 8 oz glass jar	2-6C	100 g	none
Moisture Content	SW846 3550C				
Nitrate/Nitrite	EPA Method 353.2				
Total Kjeldahl Nitrogen	SM 4500-NH3 G				
pH	SW846 9045D	4 or 8 oz glass jar	2-6C	50 g	none
Phenolics	SW846 9066	4 or 8 oz glass jar	2-6C	100 g	28 days
Phosphate	EPA Method 365.1	4 or 8 oz glass jar	2-6C	50 g	28 days
Total Phosphorus	EPA Method 365.1	4 or 8 oz glass jar	2-6C	50 g	28 days
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E				
Sulfur	SW846 6010	4 or 8 oz glass jar	2-6C	5 g	6 months
Sulfur, Reactive	SW846 7.3.4.2	4 oz glass jar	2-6C	100 g	none
Total Organic Carbon (TOC)	ASTM-2974-00				
Total Organic Carbon (TOC)	Lloyd Kahn Method	4 or 8 oz glass jar	2-6C	20 g	14 days
Total Organic Carbon (TOC)	Walkley Black Method				
Total Organic Carbon (TOC)	SW846 9060M	4 or 8 oz glass jar	2-6C	20 g	28 days
Oil and Grease	SW846-9071B	4 or 8 oz glass jar	2-6C	100 g	28 days
Pentachloraphenol	SW846-8151	4 or 8 oz glass jar	2-6C	100 g	14 days extraction/40 days analysis
Alternate Methods Provided					
PCBs	EPA Method 1668A/C	4 or 8 oz amber glass jar	2-6C	100 g	365 days
Chloride	EPA Method 300.0	4 or 8 oz glass jar	2-6C	50 g	28 days
Sulfate	EPA Method 300.0	4 or 8 oz glass jar	2-6C	50 g	28 days
Moisture Content	SM 2540G	4 or 8 oz glass jar	2-6C	50 g	none
Total Kjeldahl Nitrogen	EPA Method Method 351.2	4 or 8 oz glass jar	2-6C	50 g	28 days

O: SLM

C: SSW

F: SLM

Notes:

ASTM = ASTM International
C = degrees Celsius
CERCLA= Comprehensive Environmental Response, Compensation, and Liability Act
DI = deionized
EPA Method = Water and wastewater methods for Clean Water Act, 40 CFR 136
g = gram
MGP= Manufactured Gas Plant
mL = milliliter
OIA= OI Corporation, Published in EPA/821-R-04-001
PCB = polychlorinated biphenyl
oz = ounce
PAH = polycyclic aromatic hydrocarbon
SIM = selected ion monitoring
SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
SOP= standard operating procedure
SW-846 = EPA publication, SW-846,"Test Methods for Evaluating Solid Waste", Third Edition
USEPA or EPA= United States Environmental Protection Agency
WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
^A For list of compounds in this analytical group, see series of Tables 1 and 2
^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.
Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided
Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4B. Sampling and Analysis Summary - Pace Analytical
Soil/Sediment Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds					
Volatile Organic Compounds	SW846-8260B	1-40mL VOA vial	MeOH	10g	14 days
Volatile Organic Compounds	SW846-8021B	1-40mL VOA vial	MeOH	10g	14 days
Diesel Range Organics	WI DRO	1-4oz Clear Glass	None ^C	20-35g	10 days
Gasoline Range Organics	WI GRO	1-40mL VOA vial	MeOH	10g	14 days
Semivolatile Organic Compounds					
Semivolatile Organic Compounds	SW846 8270 SIM	1-4oz Amber Glass	None ^C	30g	14 days
Semivolatile Organic Compounds	SW846 8270 C/D	1-4oz Amber Glass	None ^C	30g	14 days
Semivolatile Organic Compounds	Alkylated, SW846-8270 MOD				
Phenols					
Phenols	SW846-8270C	1-4oz Amber Glass	None ^C	30g	14 days
Pesticides					
Chlordane	SW846 8081A	1-4oz Amber Glass	None ^C	30g	14 days
Pesticides	SW846 8081B	1-4oz Amber Glass	None ^C	30g	14 days
PCBs					
PCBs	SW846 8082A	1-4oz Amber Glass	None ^C	30g	365 days
Indicator Parameters					
Soot Carbon	none	1-4oz Amber Glass	None ^C	5g	7 days
Total Organic Carbon (TOC)	SW846 9060A	1-4oz Amber Glass	None ^C	5g	28 days
Fraction Organic Carbon (FOC)	ASTM D2974	1-4oz Amber Glass	None ^C	5g	7 days
Inorganics					
Metals	SW846-6010	1-4oz Amber Glass	None ^C	5g	6 months
Metals	SW846-6020A	1-4oz Amber Glass	None ^C	5g	6 months
Cyanide, total	SW846-9012A	1-4oz Amber Glass	None ^C	5g	14 days
Cyanide, available	OIA-1677	1-4oz Amber Glass	None ^C	10g	14 Days
Mercury	SW846-7471A/B	1-4oz Amber Glass	None ^C	5g	28 days

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Other					
Black carbon (Gustafsson)	Gustafsson Method				
Alkalinity	SM 2320B				
Ammonia	EPA Method 350.1	1-4oz Amber Glass	None ^C	5g	28 days
Biological Oxygen Demand (BOD)	SM 5210B				
British Therman Unit (BTU)	ASTM D240				
Bulk Density	ASTM D2937				
Chloride	EPA Method 325.2				
Chemical Oxygen Demand (COD)	EPA Method 410.4				
Cyanide, Reactive	SW846 7.3.3.2				
Flash Point	EPA Method 1010/1020				
Fluoride	SM 4500F-C				
Free Liquids (Paint Filter)	SW846 9095B				
Moisture Content	SW846 3550C	1-4oz poly	None ^C	25g	7 days
Nitrate/Nitrite	EPA Method 353.2	1-4oz Amber Glass	None ^C	5g	28 days
Total Kjeldahl Nitrogen	SM 4500-NH3 G				
pH	SW846 9045D	1-4oz Amber Glass	None ^C	20g	15 minutes
Phenolics	SW846 9066				
Phosphate	EPA Method 365.1				
Total Phosphorus	EPA Method 365.1				
Specific Gravity	ASTM D5057				
Sulfate	SM 4500-SO4 E				
Sulfur	SW846 6010	1-4oz Amber Glass	None ^C	5g	6 months
Sulfur, Reactive	SW846 7.3.4.2	1-4oz Amber Glass	None ^C	50g	28 days
Total Organic Carbon (TOC)	ASTM-2974-00				
Total Organic Carbon (TOC)	Lloyd Kahn Method	1-4oz Amber Glass	None ^C	5g	14 days
Total Organic Carbon (TOC)	Walkley Black Method	1-4oz Amber Glass	None ^C	5g	28 days
Total Organic Carbon (TOC)	SW846 9060M	1-4oz Amber Glass	None ^C	5g	28 days
Oil and Grease	SW846-9071B	1-4oz Amber Glass	None ^C	20g	28 days
Pentachlorophenol	SW846-8151	1-4oz Amber Glass	None ^C	30g	14 days

Table 4C. Sampling and Analysis Summary - STAT
Air/Soil Gas/Soil Vapor Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds and Indicators					
VOCs	TO-3/TO-3 Mod				
VOCs	40 CFR 60 Appendix A				
Indicator parameters	EPA Method 3C, ASTM D-1946, TO-3 Mod or 40 CFR 60 Appendix A				
Indicator parameters	EPA Method 3C				
PM-10					
PM-10/ particulate matter	PM-10 40CFR50 Appendix J	PM 10 Filter	None	1 filter	ASAP
Polycyclic Aromatic Hydrocarbons					
PAHs	TO-13A/TO-13A SIM	PUF	Cool to 0.1 - 6 C	1 PUF	7 days to Extraction, 40 days to Analysis
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-14	Summa Canister	None	1 L	30 Days
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-15	Summa Canister	None	1 L	30 Days
Volatile Organic Compounds and Polycyclic Aromatic Hydrocarbons					
VOCs and PAHs	TO-17				

O: SLM C: SSW F: SLM

Notes:
ASAP = as soon as possible
ASTM - ASTM International
C = degrees Celsius
CERCLA= Comprehensive Environmental Response, Compliance, and Liability Act
CFR = Code of Federal Regulations
MGP= Manufactured Gas Plant
MOD = modified method
L = liter
PAH = polycyclic aromatic hydrocarbon
PM₁₀ = particulate matter with a nominal diameter of 10 micrometers or less
PUF = polyurethane foam
SIM = selected ion monitoring
SOP= standard operating procedure

TO-3 = USEPA Method TO-3, Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection. See lab SOPs for additional details.
TO-13A = USEPA Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons in Ambient Air Using Gas Chromatography/Mass Spectrometry. See lab SOPs for additional details.
TO-14 = USEPA Compendium Method TO-14A, Determination Of Volatile Organic Compounds In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography. See lab SOPs for additional details.
TO-15 = USEPA Compendium method TO-15, Determination Of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry. See lab SOPs for additional details.
TO-17 = USEPA Method TO-17, Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. See lab SOPs for additional details.
USEPA or EPA = United States Environmental Protection Agency
VOC = volatile organic compound

^A For list of compounds in this analytical group, see Table 3 series
^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.
Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4C. Sampling and Analysis Summary - Test America
Air/Soil Gas/Soil Vapor Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds and Indicators					
VOCs	TO-3/TO-3 Mod				
VOCs	40 CFR 60 Appendix A				
Indicator parameters	EPA 3C	Summa Canister 6L	None	1L	30 Days
Indicator parameters	40 CFR 60 Appendix A				
PM-10					
PM-10/ particulate matter	PM-10 40CFR50 Appendix J	Glass Fiber or Quartz Filter	Cool to 0.1 - 6 C	1	None
Polycyclic Aromatic Hydrocarbons					
PAHs	TO-13A/TO-13 SIM	PUF/XAD	None	1	7 Days
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-14	Summa Canister 6L or 1L	None	1L	30 Days
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-15	Summa Canister 6L or 1L	None	1L	30 Days
Volatile Organic Compounds and Polycyclic Aromatic Hydrocarbons					
VOCs and PAHs	TO-17				

O: SLM C: SSW F: SLM

Notes:
C = Celsius
CERCLA= Comprehensive Environmental Response, Compliance, and Liability Act
CFR = Code of Federal Regulations
L = liter
MGP= Manufactured Gas Plant
MOD = modified method
PAH = polycyclic aromatic hydrocarbon
PM₁₀ = particulate matter with a nominal diameter of 10 micrometers or less
PUF = polyurethane foam
SIM = selected ion monitoring
SOP= standard operating procedure

TO-3 = USEPA Method TO-3, Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection. See lab SOPs for additional details.

TO-13A = USEPA Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons in Ambient Air Using Gas Chromatography/Mass Spectrometry. See lab SOPs for additional details.

TO-14 = USEPA Compendium Method TO-14A, Determination Of Volatile Organic Compounds In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography. See lab SOPs for additional details.

TO-15 = USEPA Compendium method TO-15, Determination Of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry. See lab SOPs for additional details.

TO-17 = USEPA Method TO-17, Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. See lab SOPs for additional details.

USEPA or EPA = United States Environmental Protection Agency
VOC = volatile organic compound
XAD = XAD resin

^A For list of compounds in this analytical group, see Table 3 series.
^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures
Sampling and analysis plan details will be confirmed with laboratory prior to field data collection

Table 4C. Sampling and Analysis Summary - Eurofins
Air/Soil Gas/Soil Vapor Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds and Indicators					
VOCs	TO-3/TO-3 Mod	(1) 1L or 6L canister per sample	None	Recommend filling canister to approximately 5 in Hg. Higher residual vacuum after collection will result in elevated reporting limits.	30 days after sample collection
VOCs	40 CFR 60 Appendix A				
Indicator parameters	EPA Method 3C, ASTM D-1946, TO-3 Mod or 40 CFR 60 Appendix A	(1) Summa Canister	None	1L	30 days
Indicator parameters	EPA Method 3C	(1) Summa Canister	None	1L	30 days
PM-10					
PM-10/ particulate matter	PM-10 40CFR50 Appendix J	(1) Filter-PM10 per sample	Do not allow filters to get wet or damaged. Do not handle with bare hands.	Method recommends sampling parameters of 0.5 m3/min for 24 hrs = 720 m3	Not specified by method. Recommended hold time of 14 days.
Polycyclic Aromatic Hydrocarbons					
PAHs	TO-13A/TO-13A SIM	Dependent on Sampling Criteria (1) High Volume or Low Volume PUF/XAD Cartridge and Quartz Filter	Ship on ice (4 C)	Low Volume: 1-5L/min for 4 to 24 hours High Volume: 0.225 m3/min for up to 24 hoursw. Up to 300 m3. Sampling volume dependent on RLs needed.	7 days from sampling to extraction.
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-14	Eurofins does not analyze samplers using TO-14 methodology, but uses TO-15 methodology for TO-14 analytical requests. See TO-15 entries.			
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-15	(1) 1L or 6L canister per sample	None	Recommend filling canister to approximately 5 in Hg. Higher residual vacuum after collection will result in elevated reporting limits.	30 days after sample collection
Volatile Organic Compounds and Polycyclic Aromatic Hydrocarbons					
VOCs and PAHs	TO-17				

O: SLM

C: SSW

F: SLM

Notes:
ASTM - ASTM International
C = Celsius
CERCLA= Comprehensive Environmental Response, Compliance, and Liability Act
CFR = Code of Federal Regulations
hr = hour
in Hg = inches mercury
L = liter
m3 = cubic meter
MGP= Manufactured Gas Plant
min = minute
MOD = modified method
PAH = polycyclic aromatic hydrocarbon
PM₁₀ = particulate matter with a nominal diameter of 10 micrometers or less
PUF = polyurethane foam
SIM = selected ion monitoring
SOP= standard operating procedure
TO-3 = USEPA Method TO-3, Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection. See lab SOPs for additional details.
TO-13A = USEPA Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons in Ambient Air Using Gas Chromatography/Mass Spectrometry. See lab SOPs for additional details.
TO-14 = USEPA Compendium Method TO-14A, Determination Of Volatile Organic Compounds In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography. See lab SOPs for additional details.
TO-15 = USEPA Compendium method TO-15, Determination Of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry. See lab SOPs for additional details.
TO-17 = USEPA Method TO-17, Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. See lab SOPs for additional details.
USEPA or EPA = United States Environmental Protection Agency
VOC = volatile organic compound
XAD = XAD resin
^A For list of compounds in this analytical group, see Table 3 series.
^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.
Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided
Sampling and analysis plan details will be confirmed with laboratory prior to field data collection.

Table 4C. Sampling and Analysis Summary - Pace Analytical
Air/Soil Gas/Soil Vapor Matrix
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Analysis/Compound Group ^A	Analytical Method Name/Number ^B	Recommended Quantity/ Container type	Preservation	Minimum Volume/Size	Holding Time
Volatile Organic Compounds and Indicators					
VOCs	TO-3/TO-3 Mod	1, Summa Canister	None	1L	30 days
VOCs	40 CFR 60 Appendix A				
Indicator parameters	EPA Method 3C, ASTM D-1946, TO-3 Mod or 40 CFR 60 Appendix A	1, Summa Canister	None	1L	30 days
Indicator parameters	EPA Method 3C	1, Summa Canister	None	1L	30 days
PM-10					
PM-10/ particulate matter	PM-10 40CFR50 Appendix J				
Polycyclic Aromatic Hydrocarbons					
PAHs	TO-13A/TO-13A SIM				
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-14				
Volatile Organic Compounds and Naphthalene					
VOCs and naphthalene	TO-15	1, Summa Canister	None	1L	30 days
Volatile Organic Compounds and Polycyclic Aromatic Hydrocarbons					
VOCs and PAHs	TO-17				

O: SLM C: SSW F: SLM

Notes:

ASTM - ASTM International

CERCLA= Comprehensive Environmental Response, Compliance, and Liability Act

CFR = Code of Federal Regulations

MGP= Manufactured Gas Plant

MOD = modified method

PAH = polycyclic aromatic hydrocarbon

PM₁₀ = particulate matter with a nominal diameter of 10 micrometers or less

SIM = selected ion monitoring

SOP= standard operating procedure

TO-3 = USEPA Method TO-3, Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection. See lab SOPs for additional details.

TO-13A = USEPA Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons in Ambient Air Using Gas Chromatography/Mass Spectrometry. See lab SOPs for additional details.

TO-14 = USEPA Compendium Method TO-14A, Determination Of Volatile Organic Compounds In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography. See lab SOPs for additional details.

TO-15 = USEPA Compendium method TO-15, Determination Of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry. See lab SOPs for additional details.

TO-17 = USEPA Method TO-17, Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. See lab SOPs for additional details.

USEPA or EPA = United States Environmental Protection Agency

VOC = volatile organic compound

^A For list of compounds in this analytical group, see Table 3 series.

^B Analytical methods name/number refer to names of analytical procedures. See lab SOPs for specific procedures.

Blank cell indicates lab is not anticipated to run these analyses, therefore data not provided

Sampling and analysis plan details will be confirmed with laboratory prior to field data collection.

Table 5. Multi-Site Program Laboratory Services - Water Analyses
Reference Sheet for Selected Labs and Range of Services
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Parameter/Analytical Method/Prep Method ^A	Laboratory								
	Pace Analytical	Test America	STAT	Brighton	Alpha	ESS	EERC	Battelle	Eurofins
GC Volatiles									
SW846-8021B	X			X					
GC/MS Volatiles									
SW846-8260B (SW846-5035, 5030, 624)	X	X	X	X	X				X
Tentatively Identified Compounds (SW846-8260) (SW846-5035, 5030, 624)	X	X	X	X	X				X
Methane, ethane, ethene (RSK175, SW846-5021, 8015B)	X	X							X
GC Semivolatiles									
SW846-8082 PCBs (SW846-3541, 3540, 3510c)	X	X	X	X	X	X			X
SW846-8081A Organochlorine Pesticides	X	X	X	X	X				X
Pesticides, select (EPA Method 1668A or C)									X
SW846-8151 Pentachlorophenol	X	X	X						X
GC/MS Semivolatiles									
SW846-8270C/D (PAHs, phenols) (SW846-3510)	X	X	X	X	X	X			X
SW846-8270C/D (PAHs, phenols, high volume injection, SW846-3510)	X	X	X	X	X				
SW846-8270C (Acid Extractables) (SW846-3510)	X	X	X	X	X	X			X
SW846-8270C (Base/Neutrals) (SW846-3510)	X	X	X	X	X	X			X
TICs (SW846-8270)	X	X	X		X	X			X
Dioxin (Low Level Method)	X	X							X
PAHs by GC-MS SIM (SW846-8270C/D)	X	X		X	X	X	X	X	X
ASTM D7363-13A (PAHs, porewater)					X		X		
Total Petroleum Hydrocarbons									
WI DRO (SW846-8270C, 8015B/C/3541/8600B/3544)	X	X		X					
WI GRO (SW846-8015C/5030B/5035A)	X	X		X					
Oil Range Organics (SW846-8270C, 8015B/C/D/5030B/5035A)		X		X	X				X
Oil & Grease, TPH (EPA Methods 1664A/B, 9071A/B)	X	X	X	X	X				X
Metals									
Single Metals by ICP (SW846-6010)	X	X		X					X
Single Metals by ICP-MS (SW846-6020, 3010/3050 extraction)	X	X	X	X	X				X
Mercury (CVAAS) (SW846-7470A, 7471)	X	X	X	X	X				X
Mercury (Low Level, SW946-7470A, 7471B, 7474)	X	X		X					X
Hexavalent Chromium (SW846-6020A, 6012, 7196A, SM3060A, SM3500Cr-B)	X	X	X	X	X				X
Trivalent Chromium (SM 3500_CR3_B)		X							
Wet Chemistry									
Alkalinity – Total (EPA Method 310.0/SM 2320B)	X	X	X	X	X				Y
Alkalinity – Carbonate/Bicarbonate (SM 2320B)	X	X	X		X				X
Biological Oxygen Demand (SM 5210B)	X	X	X	X	X				X

Parameter/Analytical Method/Prep Method ^A	Laboratory								
	Pace Analytical	Test America	STAT	Brighton	Alpha	ESS	EERC	Battelle	Eurofins
Chemical Oxygen Demand (EPA 410.4, SM 5220C))	X	X	X	X					X
Chloride (SM 4500-Cl C/E, SW846-9056A/EPA Method 300.0)	X	X	X	X					X
Available Cyanide (OIA 1677)	X	X		X					X
Total Cyanide (SW846-9012B/9014, EPA Method 335.4)	X	X	X	X	X				X
Weak Acid Dissociable Cyanide (SM 4500 CN_I)	X	X	X						X
Amenable Cyanide (SW846-9012B/9014)		X	X	X	X				X
Reactive cyanide (7.3.3.2)/ total cyanide (SW846-9012A/9014)	X	X	X	X					X
F-code solvent scan (SW846-8260B/8270D/8015)	X	X							X
Flashpoint – Closed Cup (EPA Method 1010/1020)	X	X	X	X					X
Fluoride (SM 4500F-C)	X	X	X	X					X
Free Liquids/Paint Filter (SW846 9095B)	X	X	X	X					X
Hardness as CaCO ₃ (SW846-6010/6020, SM2340/2320)	X	X	X	X					X
Ammonia (EPA Method 350.1, 210SM4500 NH ₃ _G)	X	X	X	X					X
Nitrate Nitrogen (EPA Method 353.2, SM4500, EPA Method 300.0)	X	X	X	X					X
Nitrate-Nitrite Nitrogen (EPA Method 353.2, SM4500, EPA Method 300.0)	X	X	X	X					X
Nitrate (SW946-9056)		X							
Organic Nitrogen (SM 4500-N)		X							X
Total Kjeldahl Nitrogen (EPA Method 351.2)	X	X		X					X
EPA Method 1664 Hexane Extractable Material	X	X	X	X					X
Percent moisture (ASTM D2974)	X	X	X	X					
pH (EPA Method 150.1/ SM 9040/ SM 9045)	X	X	X	X					X
Phenolics, total phenols (SW846 9066, 420.4)	X	X	X						X
Total Recoverable Phenolics (4AAP Method)	X	X		X					
Phosphate (EPA Method 365.1, SM 4500 PE)	X	X							X
Total Phosphorus (SM 4500 P_E-99)	X	X		X					X
Total Solids (SM 2540A & 2540B & 2540C & 2540D, EPA 160.4)	X	X	X	X	X				X
Total Dissolved Solids (SM 2540A & 2540B & 2540C & 2540D, EPA 160.4)	X	X	X	X	X				X
Total Suspended Solids (SM 2540A & 2540B & 2540C & 2540D, EPA 160.4)	X	X	X	X					X
Pentachlorophenol (SW846-8151)	X	X	X						X
1,2-Dibromo-3-chloropropane (DBCP) (SW846 8260C)					X				
Hexachlorocyclopentadiene (SW846 8270D)					X				
Residue, Non-filterable Total Suspended Solids (SM 2540D)	X	X	X	X					X
Sulfate (SM 4500 SO ₄ E. 300.0/375.4/9056)	X	X	X	X	X				X
Sulfide (SM4500, 9034, 9030)	X	X	X	X	X				X
Reactive sulfide (9034, 7.3.4.2, total cyanide 9034/9030M)	X	X	X	X					X
Sulfur (SW846 6010)	X	X							X
TOC (SW846 4000/9060)	X	X	X						X
TOC (SW846 5310/9060)	X	X		X					X

O: SLM C: SSW F: SLM

Notes:

4AAP = 4-aminoantipyrine

ASTM = ASTM International

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act
 CVAAS = cold vapor atomic absorption spectroscopy
 EPA Method= Water and wastewater methods for Clean Water Act, 40 CFR 136
 GC = gas chromatography
 ICP = inductively coupled plasma
 MGP = Manufactured Gas Plant
 MS = mass spectrometry
 OIA 1667 = OI Corporation, Published in EPA/821-R-04-001
 PAH = polycyclic aromatic hydrocarbon
 PCB = polychlorinated biphenyl
 RSK-175 = RSKSOP-175, 2006, prepared for the use of the Ground Water and Ecosystems Restoration Division of USEPA
 SIM = selected ion monitoring
 SM = Standard Methods for the Examination of Water and Wastewater, 20th edition
 SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition
 TIC = Tentatively identified compound
 TOC = total organic carbon
 TPH = total petroleum hydrocarbons
 USEPA or EPA = United States Environmental Protection Agency
 WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95
 WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95
 X - Lab performs analysis
^A Project-specific parameters, analytical methods and prep methods will be identified in Site-Specific Work Plans.

Table 6. Multi-Site Program Laboratory Services - Soil/Sediment Analyses
Reference Sheet for Selected Labs and Range of Services
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Parameter/Analytical Method/Prep Method ^A	Laboratory								
	Pace Analytical	Test America	STAT	Brighton	Alpha	ESS	EERC	Battelle	Eurofins
GC Volatiles									
SW846-8021B VOCs	X			X					
EnCore Samplers (SW846-5035/8021B)	X	X		X					
GC/MS Volatiles									
SW846-8260B VOCs (SW846-5035, 5030, 624)	X	X	X	X	X				X
Tentatively Identified Compounds (TICs, SW846-8260) (SW846-5035, 5030, 624)	X	X	X	X	X				X
Methane (SW846-8260) (SW846-5035, 5030, 624)	X		X	X	X				
EnCore Samplers for VOCs (SW846-8260) (SW846-5035, 5030, 624)	X	X	X	X	X				X
GC Semivolatiles									
SW846-8082 PCBs (SW846-3541, 3540)	X	X	X	X	X	X			X
SW846-8081A Organochlorine Pesticides	X	X	X	X	X	X			X
Pesticides, select (EPA Method 1668A or C)									X
SW846-8151 Pentachlorophenol (SW846-8321)	X	X	X						X
GC/MS Semivolatiles									
SW846-8270C (3546, 3510)	X	X	X	X		X			X
SW846-8270C/D (PAHs, phenols, 3546, high volume injection, 3510)	X	X	X	X					X
SW846-8270D SIM (PAHs) (3510)	X	X		X	X	X			X
SW846-8270C/D (Acid Extractables) (3510, 3520)	X	X	X	X	X	X			X
SW846-8270C/D (Base/Neutrals) (3510, 3520)	X	X	X	X	X	X			X
TICs (SW846-8270)	X	X	X	X	X	X			X
Dioxin (Low Level Method)	X	X							X
SW846-8270D SIM (Alkylated PAHs, SW846-8270-MOD)	X			X	X	X	X	X	X
Name/location of Facility that lab proposes for alkylated PAH work of forensic quality		Nashville, TN			Mansfield, MA	Cranston, RI	Grand Forks, ND	Norwell, MA	Lancaster, PA
Other forensic-related methods									
TPH with saturated hydrocarbons (SW846-8015) ¹					X	X			X
Expanded list PAHs, with homologues (GC/MS-SIM; SW846-8270D MOD) ²					X	X			X
Metals									
Single Metals by ICP (SW846-6010)	X	X		X	X				X
Single Metals by ICP/MS (SW846-6020, SW846-3010/3050 extraction)	X	X	X	X	X				X
Priority Pollutant Metal (6000/7000 series)	X	X	X	X	X				X
Mercury (SW846-7471B, 7470/ 6010, 6020)	X	X	X	X	X				X
Hexavalent Chromium	X	X	X	X	X				X
Total Petroleum Hydrocarbons									
WI DRO (SW846-8270C, 8015B/C/3541/8600B/3544)	X	X		X					
WI GRO (SW846-8015C/5030B/5035A)	X	X		X					
Oil Range Organics (SW846-8270C, 8015B/C/D/5030B/5035A)		X		X	X				X
Oil & Grease (SW846-9071B)	X	X	X		X				X
Wet Chemistry									
Alkalinity (SM 2320B)	X	X	X	X	X				
Biological Oxygen Demand (SM 5210B)	X	X	X		X				

	Laboratory								
Parameter/Analytical Method/Prep Method ^A	Pace Analytical	Test America	STAT	Brighton	Alpha	ESS	EERC	Battelle	Eurofins
British Thermal Unit (ASTM D240)		X							
Bulk Density (ASTM D2937)		X							
Chloride (EPA Method 300.0, 325.2, 9056A)	X	X	X	X					X
Chemical Oxygen Demand (EPA Method 410.4)	X		X	X					
Available Cyanide (OIA 1677)	X	X		X					X
Total Cyanide (SW846-9012B/9014/335.4)	X	X	X	X	X				X
Weak Acid Dissociable Cyanide (SM 4500CN)	X	X							X
Amenable Cyanide SW846 9012B/9014)	X	X	X	X	X				X
Digestion (SW846-3005A, 3010A, 3030c, 3050B, E 821/R-91-100)		X	X						X
F-code solvent scan (SW846-8260B/8270D/8015)		X	X	X					X
Flashpoint – Closed Cup (EPA Method 1010/1020)	X	X	X	X					X
Fluoride (SM 4500F-C, EPA Method 300.0, SW846-9056A)	X	X	X						X
Moisture Content (SW846-3550C, SM 2540G)	X	X	X						X
Ammonia (EPA Method 350.1, SM4500 NH3_G)	X	X	X	X					X
Nitrate Nitrogen (SW846-9056A/EPA Method 300.0)	X	X	X	X					X
Nitrite Nitrogen (SW846-9056A/EPA Method 300.0)	X	X	X	X					X
Nitrate-Nitrite Nitrogen (EPA Method 353.2, SM 4500NO3F)	X	X	X						X
Organic Nitrogen (SM 4500-N)		X							X
Total Kjeldahl Nitrogen (TKN) (SM 4500-NH3 G, EPA method 351.2)	X	X		X					X
EPA Method 1664 Hexane Extractable Material	X	X	X	X	X				X
Free Liquids/Paint Filter (SW846 9095B)	X	X	X	X					X
Percent moisture (ASTM D2974)	X	X	X						
Percent solid (ASTM D2216)		X	X	X					X
pH (SW846-9045C/D)	X	X	X	X					X
Phenolics, total (SW846-9066)	X	X	X						X
Soxhlet Extraction (SW846-3541)		X				X			
Total Recoverable Phenolics (4AAP Method)	X	X		X					
Phosphate (EPA Method 365.1, SM 4500P-E(M))	X	X			X				X
Total Phosphorus (EPA 365.1, SM4500 P_E-99, EPA Method 365.4)	X	X		X	X				X
Total Solids (SM2540A & 2540B & 2540C & 2540D, EPA Method 160.4)	X	X	X	X					
Specific Gravity (ASTM D5057)	X	X							X
Sulfate (SM 4500-SO4 E, SW846-9056A/EPA Method 300.0)	X	X	X						X
Sulfide (SW846-9034)	X	X	X						
Sulfur (SW846-6010)	X	X							X
Fraction organic carbon (SW846-9060A/ASTM D4129-82M/ASTM D2974/Lloyd Kahn Method)	X	X	X						
TOC (ASTM-2974-00)	X	X	X						X
TOC (Lloyd Kahn)	X	X			X				X
Black carbon (Gustafsson)		X					X		
TOC (Walkley Black)	X	X							
TOC (SW846 4000/9060)	X	X	X		X				X
Waste Characteristics									
Reactivity – Cyanide (SW846 7.3.3.2/ total cyanide SW846-9012A/9014)	X	X	X	X					X
Reactivity – Sulfide (SW846 7.3.4.2/ total sulfide SW846-9034/9030M)	X	X	X	X					X
TCLP VOCs (SW846-5035/8260/1311)	X	X	X	X	X				X
TCLP SVOCs (SW846-8270/1311)	X	X	X	X	X				X
TCLP Phenols (SW846-8270/1311)	X	X	X		X				X
TCLP Metals (SW846-1311/6010, 6020, 7470, 6000/7000 series)	X	X	X	X	X				X
TCLP organochlorine pesticides (SW846-1311/8081B)	X		X	X	X				X

Table 6

Parameter/Analytical Method/Prep Method ^A	Laboratory								
	Pace Analytical	Test America	STAT	Brighton	Alpha	ESS	EERC	Battelle	Eurofins
TCLP herbicides (SW846-1311/8151)	X		X	X	X				X
SPLP Metals/Organics (6000/7000 series)	X	X	X		X				X
ZHE TCLP Extraction	X	X	X	X	X				X
ASTM Extraction D3987	X	X	X						X
SPLP Extraction (EPA Method 1312/1311)	X	X	X		X				X
ZHE SPLP Extraction	X	X	X		X				X
Waste Management Protocol A & B	X	X	X						X
Waste Management Protocol NON-1	X	X	X						X
Other									
SPME for parent PAHs					X		X		
SPME for alkylated PAHs					X		X		

O: SLM C: SSW F: SLM

Notes:

4AAP = 4-aminoantipyrine

ASTM = ASTM International

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act

EPA Method= Water and wastewater methods for Clean Water Act, 40 CFR 136

GC = gas chromatography

ICP = inductively coupled plasma

MGP = Manufactured Gas Plant

MS = mass spectrometry

PAH = polycyclic aromatic hydrocarbon

PCB = polychlorinated biphenyl

SIM = selected ion monitoring

SM = Standard Methods for the Examination of Water and Wastewater, 20th edition

SPLP = synthetic precipitation leaching procedure

SPME = solid phase microextraction

SVOC = semivolatile organic compound

SW-846 = EPA publication, SW-846, "Test Methods for Evaluating Solid Waste", Third Edition.

TCLP = toxicity characteristic leaching procedure

TIC = tentatively identified compound

TOC = total organic carbon

TPH = total petroleum hydrocarbons

USEPA or EPA = United States Environmental Protection Agency

VOC = volatile organic compound

WI DRO = Wisconsin modified diesel range organics, WI DNR PUBL-SW-141 09/95

WI GRO = Wisconsin modified gasoline range organics, WI DNR PUBL-SW-140 09/95

X - Lab performs analysis

ZHE = zero headspace

^A Project-specific parameters, analytical methods and prep methods will be identified in Site-Specific Work Plans.

¹ This method is for TPH with saturated hydrocarbons, representing the total aromatic and aliphatic hydrocarbon content by GC/FID based on Method 8015.

² Labs indicating this capability are able to report a larger expanded list PAHs, including alkyl homologues by GC with low resolution MS using SIM. If a larger expanded list of PAHs are required for future site-specific objectives, labs will be contacted to confirm capability.

Table 7. Multi-Site Program Laboratory Services - Air/Soil Gas/Soil Vapor Analyses
Reference Sheet for Selected Labs and Range of Services
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. VW-06-C-847, VW-07-C-869, VW-07-C-877, and V-W-08-C-917

Parameter/Analytical Method/Prep Method ^A	Laboratory			
	Pace Analytical	Test America	STAT	Eurofins
Method TO-3/TO-3 Mod or 40 CFR 60 Appendix A - VOCs				
Benzene	X			X
Indicator Parameters				
EPA Method 3C, ASTM D-1946, TO-3 Mod or 40 CFR 60 Appendix A				
Methane	X	X	X	X
Carbon dioxide	X	X	X	X
Oxygen	X	X	X	X
Nitrogen	X	X	X	X
Carbon monoxide	X	X	X	X
Method PM-10 40CFR50 Appendix J				
PM-10		X	X	X
Method TO-13A - PAHs				
Naphthalene		X	X	X
Acenaphthylene		X	X	X
Acenaphthene		X	X	X
Fluorene		X	X	X
Anthracene		X	X	X
Phenanthrene		X	X	X
Fluoranthene		X	X	X
Pyrene		X	X	X
Benz(a)anthracene		X	X	X
Chrysene		X	X	X
Benzo(b)fluoranthene		X	X	X
Benzo(k)fluoranthene		X	X	X
Benzo(a)pyrene		X		X
Benzo(g,h,i)perylene		X	X	X
Indeno(1,2,3-cd)pyrene		X	X	X
Dibenz(a,h)anthracene		X	X	X
Method TO-14 - VOCs and Naphthalene		see footnote B		see footnote B
1,1,2,2-Tetrachloroethane		X	X	X
1,1,2-Trichloroethane (Vinyl trichloride)		X	X	X
1,1-Dichloroethane (Ethylidene chloride)		X	X	X
1,2,4-Trichlorobenzene		X	X	X
1,2,4-Trimethylbenzene (Pseudocumene)		X	X	X
1,2-Dibromoethane (Ethylene dibromide)		X	X	X
1,2-Dichloroethane (Ethylene dichloride)		X	X	X
1,2-Dichloropropane (Propylene dichloride)		X	X	X
1,3,5-Trimethylbenzene (Mesitylene)		X	X	X
Benzene (Cyclohexatriene)		X	X	X
Benzyl chloride (cx-Chlorotoluene)		X	X	X
Carbon tetrachloride (Tetrachloromethane)		X	X	X
Chlorobenzene (Phenyl chloride)		X	X	X

Parameter/Analytical Method/Prep Method ^A	Laboratory			
	Pace Analytical	Test America	STAT	Eurofins
Chloroform (Trichloromethane)		X	X	X
cis-1,2-Dichloroethylene		X	X	X
cis-1,3-Dichloropropene (cis-1,3- dichloropropylene)		X	X	X
Dichloromethane (Methylene chloride)		X	X	X
Ethyl chloride (Chloroethane)		X	X	X
Ethylbenzene		X	X	X
Freon 11 (Trichlorofluoromethane)		X	X	X
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)		X	X	X
Freon 114 (1,2-Dichloro-1,1,2,2- tetrafluoroethane)		X	X	X
Freon 12 (Dichlorodifluoromethane)		X	X	X
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro- 1,3-butadiene)		X	X	X
m-Dichlorobenzene (1,3-Dichlorobenzene)		X	X	X
Methyl bromide (Bromomethane)		X	X	X
Methyl chloride (Chloromethane)		X	X	X
Methyl chloroform (1,1,1-Trichloroethane)		X	X	X
m-Xylene (1,3-Dimethylbenzene)		X	X	X
Naphthalene		X		X
o-Dichlorobenzene (1,2-dichlorobenzene)		X	X	X
o-Xylene (1,2-Dimethylbenzene)		X	X	X
p-Dichlorobenzene (1,4-dichlorobenzene)		X	X	X
p-Xylene (1,4-Dimethylxylene)		X	X	
Styrene (Vinyl benzene)		X	X	X
Tetrachloroethylene (Perchloroethylene)		X	X	X
Toluene (Methyl benzene)		X	X	X
trans-1,3-Dichloropropene (trans-1,3- Dichloropropylene)		X	X	X
Trichloroethylene (Trichloroethene)		X	X	X
Vinyl chloride (Chloroethylene)		X	X	X
Vinylidene chloride (1,1-Dichloroethene)		X	X	X
Method TO-15 - VOCs and Naphthalene				
1,1,2-Trichloroethane; C2H3Cl3	X	X	X	X
1,2,4-Trichlorobenzene; C6H3Cl3	X	X	X	X
1,3-Butadiene; C4H6	X	X	X	X
1,3-Dichloropropene; C3H4Cl2 (cis)		X	X	X
1,4-Dichlorobenzene (p-); C6H4Cl2	X	X	X	X
1,4-Dioxane (1,4-Diethylene oxide); C4H8O2	X	X	X	X
2,2,4-Trimethyl pentane C8H18	X	X		X
Acetonitrile (cyanomethane); C2H3N		X		
Acrolein (2-propenal); C3H4O	X	X		
Acrylonitrile (2-propenenitrile); C3H3N	X	X		
Allyl chloride (3-chloropropene); C3H5Cl	X	X		
Benzene; C6H6	X	X	X	X
Benzyl chloride (a-chlorotoluene); C7H7Cl	X	X	X	X
Bromoform (tribromomethane); CHBr3	X	X	X	X
Carbon disulfide; CS2	X	X	X	X
Carbon tetrachloride; CCl4	X	X	X	X
Chlorobenzene; C6H5Cl	X	X	X	X
Chloroform; CHCl3	X	X	X	X
Cumene (isopropylbenzene); C9H12	X	X		X
Ethyl chloride (chloroethane); C2H5Cl	X	X	X	X
Ethylbenzene; C8H10	X	X	X	X
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	X	X	X	X

Table 7

Parameter/Analytical Method/Prep Method ^A	Laboratory			
	Pace Analytical	Test America	STAT	Eurofins
Ethylene dichloride (1,2-dichloroethane); C ₂ H ₄ Cl ₂	X	X	X	X
Ethylidene dichloride (1,1-dichloroethane); C ₂ H ₄ Cl ₂	X	X	X	X
Hexachlorobutadiene; C ₄ Cl ₆	X	X	X	X
Hexane; C ₆ H ₁₄	X	X	X	X
1,1,2,2-Tetrachloroethane; C ₂ H ₂ Cl ₄	X	X	X	X
Methyl bromide (bromomethane); CH ₃ Br	X	X	X	X
Methyl chloride (chloromethane); CH ₃ Cl	X	X	X	X
Methyl chloroform (1,1,1-trichloroethane); C ₂ H ₃ Cl ₃	X	X		X
Methyl ethyl ketone (2-butanone); C ₄ H ₈ O	X	X	X	X
Methyl isobutyl ketone (hexone); C ₆ H ₁₂ O	X	X		X
Methyl methacrylate; C ₅ H ₈ O ₂	X			
Methyl tert-butyl ether; C ₅ H ₁₂ O	X	X	X	X
Methylene chloride; CH ₂ Cl ₂	X	X	X	X
m-Xylene; C ₈ H ₁₀	X	X	X	X
Naphthalene	X	X	X	X
o-Xylene; C ₈ H ₁₀	X	X	X	X
Propylene dichloride (1,2-dichloropropane); C ₃ H ₆ Cl ₂	X	X	X	X
p-Xylene; C ₈ H ₁₀	X	X	X	
Styrene; C ₈ H ₈	X	X	X	X
Tetrachloroethylene; C ₂ Cl ₄	X	X	X	X
Toluene; C ₇ H ₈	X	X	X	X
Trichloroethylene; C ₂ HCl ₃	X	X	X	X
Vinyl acetate; C ₄ H ₆ O ₂	X	X	X	
Vinyl bromide (bromoethene); C ₂ H ₃ Br	X	X		
Vinyl chloride (chloroethene); C ₂ H ₃ Cl	X	X	X	X
Vinylidene chloride (1,1-dichloroethylene); C ₂ H ₂ Cl ₂	X	X	X	X
Xylenes (isomer & mixtures); C ₈ H ₁₀	X	X	X	
Method TO-17 - VOCs and PAHs				
1,1,1,2-Tetrachloroethane				X
1,1,1-Trichloroethane				X
1,1,2,2-Tetrachloroethane				X
1,1,2-Trichloroethane				X
1,2,3-Trimethylbenzene				X
1,2,3-Trimethylbenzene				X
1,2-Dichloroethane				X
1,3,5-Trimethylbenzene				X
1-Methyl-3-ethylbenzene				X
3,5,5-Trimethylcyclohex-2-enone				X
Acetic acid				X
Acetone				X
Acetonitrile				X
Acrylonitrile				X
all Xylenes				X
Aniline				X
Benzene				X
Butoxyethanol				X
Butoxyethylacetate				X
Butylacetate				X
Carbontetrachloride				X
Chlorobenzene				X
Cyclohexanone				X
Decane				X

Table 7

Parameter/Analytical Method/Prep Method ^A	Laboratory			
	Pace Analytical	Test America	STAT	Eurofins
Dichloromethane				X
Ethanol				X
Ethoxyethanol				X
Ethoxyethylacetate				X
Ethylacetate				X
Ethylacrylate				X
Ethylbenzene				X
Furfural				X
iso-Butanol				X
Isobutylacetate				X
Isopropanol				X
Isopropylacetate				X
Isopropylbenzene				X
Maleic anhydride				X
Methanol				X
Methoxyethanol				X
Methoxyethylacetate				X
Methoxypropanol				X
Methyl-2-ethylbenzene				X
Methyl-4-ethylbenzene				X
Methylacetate				X
Methylacrylate				X
Methylethylketone (2-butanone)				X
Methylisobutylketone				X
Methylmethacrylate				X
Methylstyrene				X
Methyl-t-butyl ether				X
n-Butanal				X
n-Butane				X
n-Butanol				X
n-Dodecane				X
n-Heptane				X
n-Hexane				X
Nitrobenzene				X
n-Nonane				X
n-Octane				X
n-Pentane				X
n-Propanol				X
n-Propylbenzene				X
n-Undecane				X
Octanol				X
Phenol				X
Propionitrile				X
Propylacetate				X
Pyridine				X
Styrene				X
t-Butylacetate				X
Tetrachloroethylene				X
Toluene				X
Trichloroethylene				X

O: SLM C: SSW F: SLM

Table 7

Notes:

ASTM = ASTM International

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act

CFR = Code of Federal Regulations

MGP = Manufactured Gas Plant

PAH = polycyclic aromatic hydrocarbon

PM₁₀ = particulate matter with a nominal diameter of 10 micrometers or less

SOP = standard operating procedure

TO-3 = USEPA Method TO-3, Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection. See lab SOPs for additional details.

TO-13A = USEPA Compendium Method TO-13A, Determination of Polycyclic Aromatic Hydrocarbons in Ambient Air Using Gas Chromatography/Mass Spectrometry. See lab SOPs for additional details.

TO-14 = USEPA Compendium Method TO-14A, Determination Of Volatile Organic Compounds In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography. See lab SOPs for additional details.

TO-15 = USEPA Compendium method TO-15, Determination Of Volatile Organic Compounds In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry. See lab SOPs for additional details.

TO-17 = USEPA Method TO-17, Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. See lab SOPs for additional details.

USEPA or EPA = United States Environmental Protection Agency

VOC = volatile organic compound

X - Lab performs analysis

^A Project-specific parameters, analytical methods and prep methods will be identified in Site-Specific Work Plans.

^B Eurofins does not analyze samples using TO-14 methodology, but uses TO-15 methodology for TO-14 analytical requests. TO-15 in the newer air method for VOC analysis and meets the TO-14 requirements. Test America also prefers TO-15, but can do TO-14.

Table 8. Data Measurement Units for Field and Laboratory Measurements
MGP Multi-Site Program
USEPA Region 5
CERCLA Docket Nos. V-W-'06-C-847, V-W-'07-C-869, and V-W-'07-C-877

Parameter	Units
pH	pH units or standard units
Temperature	degrees Celsius (°C)
Turbidity	Nephelometric Turbidity Unit (NTU)
Dissolved Oxygen	parts per million (ppm) or milligrams per liter (mg/L)
Specific Conductance	microsiemens per centimeter at 25°C (uS/cm)
Concentration of chemical in water matrix	micrograms per liter (ug/L) organic milligrams per liter (mg/L) inorganic
Concentration of chemical in soil/sediment matrix	micrograms per kilogram (ug/kg) milligrams per kilogram (mg/kg)
Concentration of chemical in air soil gas/vapor matrix	micrograms per cubic meter (ug/m ³)
Organic Content by Loss-on-Ignition	percent (%)
Total Organic Carbon	milligrams per kilogram (mg/kg)
Atterberg Limits	percent (%)
Grain Size Distribution	percent (%)
Specific Gravity	(dimensionless)
Moisture Content	percent (%)
Strength	tons per square foot (tsf)

Notes:

CERCLA= Comprehensive Environmental Response, Compensation and Liability Act

MGP= manufactured gas plant

USEPA= United States Environmental Protection Agency



Enclosures



**Enclosures A1-A10
Laboratory/Validators
Documentation
(Electronic Files)**



Enclosure B
Multi-Site QAPP
Addendum No. 1

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Title: Multi-Site QAPP**Addendum****Addendum Date: 3/12/12****Page _1_ of _2_****QC Samples Table**

Matrix	Aqueous and Non-Aqueous
Analytical Group	VOCs/SVOCs
Concentration Level	All
Sampling SOP	SAS-08-02/SAS-06-01
Analytical Method/ SOP Reference	SW846 8260/8270
Sampler's Name	TBD
Field Sampling Organization	TBD
Analytical Organization	TBD
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field duplicate (aqueous)	1 in 10 investigative samples, unless otherwise specified	NA	Estimate (J) positive values	Project manager, with data validator and laboratory manager, as needed	Calculate RPD for compounds detected at concentrations $\geq 2x$ the quantitation limit (QL)	RPD<30%, per Multi-Site QAPP Table 6 and Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Revised (USEPA, December 1996)
			Use professional judgement to accept, qualify or reject positive detects for the compound. If data is rejected, location may be re-sampled.		Calculate RPD for compounds detected at concentrations \geq QL and $< 2x$ QL	
Field duplicate (non-aqueous)	1 in 20 investigative samples, unless otherwise specified		Estimate (J) positive values		Calculate RPD for compounds detected at concentrations $\geq 2x$ QL	
			Use professional judgement to accept, qualify or reject positive detects for the compound. If data is rejected, location may be re-sampled.		Calculate RPD for compounds detected at concentrations \geq QL and $< 2x$ QL	

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Title: Multi-Site QAPP**Addendum****Addendum Date: 3/12/12****Page _1_ of _2_****QC Samples Table**

Matrix	Aqueous and Non-Aqueous
Analytical Group	Inorganics
Concentration Level	All
Sampling SOP	SAS-08- 02/SAS-06- 01
Analytical Method/ SOP Reference	SW846 6020/7471A/ 9012A/OIA 1677
Sampler's Name	TBD
Field Sampling Organization	TBD
Analytical Organization	TBD
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field duplicate (aqueous)	1 in 10 investigative samples, unless otherwise specified	NA	Estimate (J) positive values	Project manager, with data validator and laboratory manager, as needed	Calculate RPD for compounds detected at concentrations $\geq 5x$ the quantitation limit (QL)	RPD<30%, per Multi-Site QAPP Table 6 and Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Revised (USEPA, December 1996)
					Calculate absolute difference for compounds detected at concentrations $< 5x$ QL	
Field duplicate (non-aqueous)	1 in 20 investigative samples, unless otherwise specified				Calculate RPD for compounds detected at concentrations $\geq 5x$ QL	
					Calculate absolute difference for compounds detected at concentrations $< 5x$ QL	

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Title: Multi-Site QAPP**Addendum****Addendum Date: 3/12/12****Page _1_ of _2_****QC Samples Table**

Matrix	Aqueous and Non-Aqueous
Analytical Group	PCBs
Concentration Level	All
Sampling SOP	SAS-08-02/SAS-06-01
Analytical Method/ SOP Reference	SW846 8081
Sampler's Name	TBD
Field Sampling Organization	TBD
Analytical Organization	TBD
No. of Sample Locations	TBD

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field duplicate (aqueous)	1 in 10 investigative samples, unless otherwise specified	NA	Estimate (J) positive values	Project manager, with data validator and laboratory manager, as needed	Calculate RPD for compounds detected at concentrations $\geq 2x$ the quantitation limit (QL)	RPD<30%, per Multi-Site QAPP Table 6 and Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Revised (USEPA, December 1996)
			Use professional judgement to accept, qualify or reject positive detects for the compound. If data is rejected, location may be re-sampled.		Calculate RPD for compounds detected at concentrations \geq QL and $< 2x$ QL	
Field duplicate (non-aqueous)	1 in 20 investigative samples, unless otherwise specified		Estimate (J) positive values		Calculate RPD for compounds detected at concentrations $\geq 2x$ QL	
			Use professional judgement to accept, qualify or reject positive detects for the compound. If data is rejected, location may be re-sampled.		Calculate RPD for compounds detected at concentrations \geq QL and $< 2x$ QL	



Enclosure C
Multi-Site QAPP
Addendum No. 2



ENVIRONMENTAL CONSULTANTS

300 S. WACKER DRIVE, SUITE 2050
CHICAGO, IL 60606
(P) 312.465.1740
(F) 262.523.9001

Mr. Ross del Rosario
USEPA Region 5 – SR6J
77 W. Jackson Boulevard
Chicago, Illinois 60604-3590

May 7, 2012
Project No. 2037

RE: Updated Laboratory Usage and Qualification Addendum– TestAmerica Laboratories
Former Crawford Station MGP Site, Chicago, Illinois
Peoples Gas Light and Coke Company

CERCLA Docket No. V-W-08-C-917 and V-W-11-C-981
CERCLIS ID – ILN0000510192

Dear Mr. del Rosario:

On behalf of Integrys Business Support, LLC (IBS), Natural Resource Technology, Inc. (NRT) is providing this notice for the proposed laboratory for soil and air samples at the Former Crawford Station Manufactured Gas Plant (MGP) Site. This notification will serve as addendum to the IBS Multi-Site Quality Assurance Project Plan (QAPP) and provides the rationale for use of TestAmerica Laboratories, Inc. as well as the pertinent sheets from the Workbook for Uniform Federal Policy (UFP) for Quality Assurance Plans. This laboratory will be used for the Removal Action and the future Remedial Investigation/Feasibility Study (RI/FS) activities that are planned for the site.

TestAmerica was cited in the Removal Action Work Plan (RAWP) (Revision 1 dated September 6, 2011) as one of the laboratories that would be used for soil and air sample analysis. However, the approved Multi-Site QAPP prepared for IBS (Revision 2) did not include TestAmerica for all of the chemical constituents/media that require laboratory analysis for this project.

TestAmerica is an accredited laboratory under the National Environmental Laboratory Accreditation Program (NELAP) and therefore meets the quality system criteria specified under Paragraph 18 of AOC V-W-11-C-981 pertaining to the Time Critical Removal Action work. However, in order to assure the reliability of the soil data for the future RI/FS to be performed pursuant to AOC V-W-08-C-917, we are seeking to expand the scope of TestAmerica's approval under the Multi-Site QAPP so that it is deemed qualified to perform a full range of soil analytical procedures as well as perform analysis of air samples for the Former Crawford Station MGP Site. Included in this notification are the pertinent UFP QAPP worksheets and Standard Operating Procedures (SOP) detailing laboratory procedures and analytical specifications.

Rationale for Use of TestAmerica Laboratories

- Due to TestAmerica's location in University Park, Illinois, access to the laboratory will reduce the turn-around time for sample analysis resulting in greater efficiency of site activities.
- TestAmerica has a larger capacity to handle expedited sample analysis than most laboratories located in the greater Chicagoland area. This is expected to result in fewer delays in the reporting of analytical results and facilitate greater efficiency in the execution of site excavation and backfilling activities.
- TestAmerica analytical procedures meet the anticipated action levels for IBS projects. This will result in data which can be applied to both the Time Critical Removal Action AOC as well as the RI/FS AOC.
- Utilizing TestAmerica's Burlington, Vermont and Los Angeles, California air laboratories will allow the soil and air samples to be picked up concurrently to simplify sample shipping and handling procedures.

WWW.NATURALRT.COM

Laboratory Requirements

Laboratory-specific UFP QAPP worksheets have been completed and are included with this notification as Attachment 1. Laboratory-specific Standard Operating Procedures (SOPs) have been included with this notification as Attachment 2. Laboratory-specific procedures and performance data meet the analytical requirements for this work based on the risk-based Screening Levels previously outlined in the approved Risk Assessment Framework (Exponent, 2011).

We request your approval of TestAmerica for the purposes indicated herein.

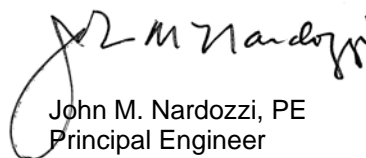
Please contact Mr. Naren Prasad of IBS at 312.240.4569 if you should have any questions regarding the content of this submittal.

Sincerely,

NATURAL RESOURCE TECHNOLOGY, INC.



Timothy B. Norris, PG
Geologist



John M. Nardozzi, PE
Principal Engineer

Attachments:

Attachment 1 – UFP QAPP Worksheets

QAPP Worksheet #1	Title and Approval Page
QAPP Worksheet #2	QAPP Identifying Information
QAPP Worksheet #6	Communication Pathways
QAPP Worksheet #9	Project Scoping Session Participants Sheet
QAPP Worksheet #15	Reference Limits and Evaluation Table
QAPP Worksheet #19	Analytical SOP Requirements Table
QAPP Worksheet #23	Analytical SOP Reference Table
QAPP Worksheet #24	Analytical Instrument Calibration Table
QAPP Worksheet #25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
QAPP Worksheet #30	Analytical Services Table

Attachment 2 – Laboratory Specific Data Submittals

UP-QA-QAM	Quality Assurance Manual, 03/03/11, Rev. 3
UP-MV-8260	Gas Chromatography Mass Spectrometry-Volatiles, 11/20/11, Rev 22
UP-MB-8270C	Gas Chromatography Mass Spectrometry-Semi-Volatiles, SW846 Method EPA 8270C, 11/18/11, Rev 20
UP-GE-DRO	Gas Chromatography: Semi-Volatiles Diesel Range Organics (DRO), 09/30/2011, Rev 15
UP-SP-3541	Sample Preparation Semivolatile and Nonvolatile Organic Compounds from a Soil/Sediment Matrix using Soxhlet Extraction, 10/03/2011, Rev 10
UP-GV-GRO	Gas Chromatography: Volatiles Gasoline Range Organics (GRO), 09/30/2011, Rev 15
UP-ME-6010B	Metals Analysis Trace Inductively Coupled Argon Plasma by SW846 6010B (Simultaneous Operation), 09/30/2011, Rev 14
UP-SP-3000	Sample Preparation Metals Digestion by SW-846 3000 Series, 03/03/2011, Rev 20
UP-ME-245.1	Metals Analysis Mercury by EPA Methods 245.1/245.5; SW-846 7470A/7471A/7471B; and U.S. EPA CLP Doc No ILM04.0, 10/28/11, Rev 17
UP-WC-CN	Wet Chemistry Cyanide (Total/Weak Acid Dissociable/Amenable/Reactive), 10/28/2011, Rev 23
UP-WC-Sulfide	Wet Chemistry Total Acid Soluble, Acid-Volatile and Reactive Sulfide, 11/01/2010, Rev 15
BR-AT-004	Determination of VOCs in Ambient Air (EPA Compendium Method TO15), BR-AT-004, 09/25/09, Rev 7
LA-MSA-151	Determination of Low-Level VOCs in Ambient/Indoor Whole Air Samples using GC/MS-SIM Mode, LA-MSA-151, 12/11/09, Rev 6
BR-AT-002	Determination of Carbon Dioxide (CO ₂), Oxygen (O ₂), Nitrogen (N ₂), Methane (CH ₄) and Carbon Monoxide (CO) By GC/TCD (EPA Method 3C), 11/01/11, Rev 2

cc: Mr. Doyle. Wilson, IEPA (via US Mail and email)
Mr. Naren Prasad, IBS (via email)
Mr. David Klatt, CH2M Hill (via email)
Ms. Jennifer Kahler, NRT

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QAPP Worksheet #2.	QAPP Identifying Information	6
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QAPP Worksheet #9.	Project Scoping Session Participants Sheet	12
QAPP Worksheet #15.	Reference Limits and Evaluation Table	14
QAPP Worksheet #19.	Analytical SOP Requirements Table	28
QAPP Worksheet #23.	Analytical SOP Reference Table	29
QAPP Worksheet #24.	Analytical Instrument Calibration Table	30
QAPP Worksheet #25.	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	33
QAPP Worksheet #30.	Analytical Services Table	34

Title: Supplemental Multi-Site QAPP

Revision Number: 0

Revision Date: 5/3/12

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**QAPP Worksheet #1
(UEP-QAPP Manual Section 2.1)
Title and Approval Page**

Site Name/Project Name: Crawford Station Former MGP Time Critical Removal Action
Site Location: Chicago, Illinois

Document Title: Supplemental Multi-Site QAPP Worksheets

Lead Organization: Integrys Business Support, LLC

Preparer's Name and Organizational Affiliation: Timothy Norris, NRT

Preparer's Address, Telephone Number, and E-mail Address: 311 S. Wacker Drive, Suite 1670
Chicago, Illinois 60606
312-465-1740 ext 2105
tnorris@naturalrt.com

Preparation Date (Day/Month/Year): 9/April/2012

Lead Organization's Project Manager: _____

Signature

Printed Name/Organization/Date: Naren Prasad, IBS

Investigative Organization's Project Manager: _____

Signature

Printed Name/Organization/Date: John Nardozi, NRT

Investigative Organization's Project QA Officer: _____

Signature

Printed Name/Organization/Date: Jennifer Kahler, NRT

Approval Signature: _____

Signature

Printed Name/Organization/Date: Ross del Rosario USEPA Region V Project Coordinator

Approval Signature: _____

Signature

Printed Name/Organization/Date: Alida Roberman, USEPA Region V QA Coordinator

Document Control Number: _____

QAPP Worksheet #2
(UFP-QAPP Manual Section 2.2.4)
QAPP Identifying Information

Site Name/Project Name: Crawford Station Former MGP Time Critical Removal Action

Site Location: Chicago, Illinois

Site Number/Code: ILN000510192

Operable Unit: Crawford Station

Contractor Name: Natural Resource Technology

Contractor Number: Not Applicable

Contract Title: Not Applicable

Work Assignment Number: Not Applicable

1. Identify guidance used to prepare QAPP: USEPA Guidance for QAPPs and UFP Worksheets

2. Identify regulatory program: CERCLA Superfund Alternative Sites Program

3. Identify approval entity: Not Applicable

4. Indicate whether the QAPP is a generic or a project-specific QAPP. Generic for use on Crawford Time Critical Removal Action

5. List dates of scoping sessions that were held: 8/17/2011, 1/24/12

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title: Multi-Site QAPP - Quality Assurance Project Plan, Former Manufactured Gas Plant Sites, Volume 1 and 2, Revision 2 (IBS, September 2007)

Approval Date: 12/5/2007

7. List organizational partners (stakeholders) and connection with lead organization: _____

8. List data users: Integrays Business Support, LLC (IBS), Natural Resource Technology, Inc. (NRT), Exponent, Burns & McDonnell (BMCD), and USEPA Region V

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below: _____

Documents referenced in table below are.

AOC V-W-11-C-981- *Administrative Settlement Agreement and Order on Consent for Removal Action, Crawford Station (USEPA, September 2011)*

Multi-Site QAPP - *Quality Assurance Project Plan, Former Manufactured Gas Plant Sites, Volume 1 and 2, Revision 2 (IBS, September 2007)*

Multi-Site FSP – *IBS 2008, Multi-Site Field Sampling and Analysis Plan, Former Manufactured Gas Plant Sites, Volume 1 and 2, Revision 4 (IBS, September 2008)* _

QAPP Worksheet #2
QAPP Identifying Information
Continued

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Worksheet #1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	Worksheet #2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Multi-Site QAPP Distribution List and Sign-off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	Multi-Site QAPP Section 1.2 and Figure 1 Worksheet #6
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	AOC V-W-11-C-981 or as specified in Site Specific Work Plan (project scoping) Worksheet #9 Multi-Site QAPP Section 1.3 (problem definition and background)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	Multi-Site QAPP Section 1.5 (quality objectives) Risk Assessment Framework Addendum, April 2011 (Exponent) or as specified in Site Specific Work Plans
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	Upcoming Site Specific Work Plan
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Multi-Site QAPP Section 1.5 (quality objectives) or as specified in Site Specific Work Plan
Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/ SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Multi-Site QAPP Section 2. and Multi-Site FSP Section 4 2 (field sampling SOPs) Multi-Site QAPP Section 2.2.3 (field equipment calibration) Multi-Site QAPP Section 2.2.4 (supply inspection and acceptance) Multi-Site QAPP Section 2.9.1 (field documentation); Multi-Site FSP Appendix B (field forms) or as specified in Site Specific Work Plan

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Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Worksheet #23, #24, and #25
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	Multi-Site QAPP Section 2.3 and Attachment 1 Multi-Site FSP section 5
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	Multi-Site QAPP Section 2.5 or as specified in Site Specific Work Plan
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	Multi-Site QAPP Section 2.3.2.3 Multi-Site FSP Section 4.9
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	Multi-Site QAPP Section 3.1
4.2 QA Management Reports	- QA Management Reports Table	Multi-Site QAPP Section 3.2.1
4.3 Final Project Report		Multi-Site QAPP Section 3.2.2 and 3.2.3
Data Review		
5.1 Overview		

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	AOC V-W-11-C-9Multi-Site QAPP Section 4
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		

QAPP Worksheet #6

(UFP-QAPP Manual Section 2.4.2)

Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modification to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Change to sampling plan (sample locations, numbers, or analytes) due to field conditions	Natural Resource Technology, Inc	Timothy Norris, or designee	312-465-1740 ext 2105	Request approval from USEPA prior to authorizing sample collection or laboratory analysis

QAPP Worksheet #9

(UFP-QAPP Manual Section 2.5.1)

Complete this worksheet for each project scoping session held.

Identify project team members who are responsible for planning the project.

Project Scoping Session Participants Sheet

Project Name: Removal Action				Site Name: Crawford Station MGP Site	
Projected Date(s) of Sampling: 1/2012 – 12/2012				Site Location: 3500 S. Pulaski, Chicago, IL	
Project Manager: John M. Nardoizzi, P.E.					
Date of Session: 8/17/2011					
Scoping Session Purpose: Discussion of Removal Action Plan					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Ross del Rosario	RPM	USEPA Reg. 5	312 886 6195	Delrosario.rosauro@epamail.epa.gov	PGL Sites RPM
Peter Felitti	ORC	USEPA Reg. 5	312 886 5114	Felitti.peter@epa.gov	Legal Counsel
David Klatt	USEPA Technical Consultant	CH ₂ M Hill	773 458 2832	dklatt@ch2m.com	Technical consultant to USEPA
Naren Prasad	Sr. Env. Engineer	IBS	312 240 4569	nmprasad@integrys-group.com	IBS Project Coordinator
John M. Nardoizzi	Principal Engineer	NRT	312 465 1740 ext 2102	jnardoizzi@narturalr-t.com	Crawford Project Manager

Comments/Decisions: Review of Removal Action Work Plan (RAWP), Rev. 0

- Discussion of sampling frequency for overburden samples
- Review of sample parameters for overburden samples and post-excavation samples

Action Items: Revise and reissue RAWP as Revision 1 to incorporate USEPA Comments.

Consensus Decisions: _____

QAPP Worksheet #9

(UFP-QAPP Manual Section 2.5.1)

Complete this worksheet for each project scoping session held.

Identify project team members who are responsible for planning the project.

Project Scoping Session Participants Sheet

Project Name: RI/FS				Site Name: Crawford Station MGP Site	
Projected Date(s) of Sampling: 2012 and beyond				Site Location: 3500 S. Pulaski, Chicago, IL	
Project Manager: John M. Nardoizzi, P.E.					
Date of Session: 1/24/2012					
Scoping Session Purpose: Discussion of Site Specific Work Plans (SSWPs)					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Ross del Rosario	RPM	USEPA Reg. 5	312 886 6195	Delrosario.rosario@epamail.epa.gov	PGL Sites RPM
David Klatt	USEPA Technical Consultant	CH ₂ M Hill	773 458 2832	dklatt@ch2m.com	Technical consultant to USEPA
Naren Prasad	Sr. Env. Engineer	IBS	312 240 4569	nmprasad@integralsgroup.com	IBS Project Coordinator
John M. Nardoizzi	Principal Engineer	NRT	312 465 1740 ext 2102	jnardoizzi@narturalrt.com	Crawford Project Manager

Comments/Decisions: Pre-scoping meeting for Site Specific Work Plan, Rev. 0

- Discussion of proposed phasing plan for multiple SSWPs for entire site
- Discussion of potential access issues related to site due to multiple property owners

Action Items: Proceed with preparation of first SSWP (i.e., Phase I) to address Parcels Q, R, O, P and K.

Consensus Decisions: Phasing of the SSWPs with four phases was appropriate based on the size, complexity and access constraints related to the Crawford site.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: TCLP

Analytical Group: VOC

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/L)	Project Quantitation Limit (mg/L)	EPA 8260B/EPA 5035A ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/L)	QLs (mg/L)
Benzene	71-42-2	0.5	0.34	Not Listed	Not Listed	0.01	0.02

¹Analytical MDLs and QLs are those documented in validated methods.²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.**Lab Notes: Reporting Limits and Method Detection Limits are subject to change.**

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: VOC

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 8260B/EPA 5035A ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Benzene	71-43-2	0.255	0.255	Not Listed	Not Listed	0.00054	0.005
Ethylbenzene	100-41-4	5.16	5.16	Not Listed	Not Listed	0.00075	0.005
Toluene	108-88-3	5.45	5.45	Not Listed	Not Listed	0.00097	0.005
Xylenes, Total	1330-20-7	10	10	Not Listed	Not Listed	0.0007	0.01
1,2,4-Trimethylbenzene	95-63-6	62	62	Not Listed	Not Listed	0.00074	0.005
1,3,5-Trimethylbenzene	108-67-8	780	780	Not Listed	Not Listed	0.00104	0.005
Bromoform	75-25-2	15.9	15.9	Not Listed	Not Listed	0.00081	0.005
Carbon tetrachloride	56-23-5	0.61	0.61	Not Listed	Not Listed	0.00109	0.005
Chlorobenzene	108-90-7	13.1	13.1	Not Listed	Not Listed	0.00079	0.005
Chloroform	67-66-3	0.29	0.29	Not Listed	Not Listed	0.00092	0.005
1,2-Dichloroethane	107-06-2	0.43	0.43	Not Listed	Not Listed	0.00051	0.005
1,1-Dichloroethene	75-35-4	8.28	8.28	Not Listed	Not Listed	0.00079	0.005
cis-1,2-Dichloroethene	156-59-2	160	160	Not Listed	Not Listed	0.00073	0.005
trans-1,2-Dichloroethene	156-60-5	0.784	0.784	Not Listed	Not Listed	0.00071	0.005
Dichlorobromomethane	75-27-4	0.68	0.68	Not Listed	Not Listed	0.00076	0.005
Dichloromethane	75-09-2	4.05	4.05	Not Listed	Not Listed	0.0014	0.005
1,2-Dichloropropane	78-87-5	0.89	0.89	Not Listed	Not Listed	0.00113	0.005
cis-1,3-Dichloropropene	10061-01-5	0.398	0.398	Not Listed	Not Listed	0.00057	0.005
trans-1,3-Dichloropropene	10061-02-6	0.398	0.398	Not Listed	Not Listed	0.00113	0.005
Styrene	100-42-5	4.69	4.69	Not Listed	Not Listed	0.00063	0.005
Tetrachloroethene	127-18-4	0.55	0.55	Not Listed	Not Listed	0.00095	0.005
1,1,1-Trichloroethane	71-55-6	29.8	29.8	Not Listed	Not Listed	0.00096	0.005

Title: Supplemental Multi-Site QAPP

Revision Number: 0

Revision Date: 5/3/12

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1,1,2-Trichloroethane	79-00-5	1.1	1.1	Not Listed	Not Listed	0.00067	0.005
Trichloroethene	79-01-6	2.8	2.8	Not Listed	Not Listed	0.00081	0.005
Vinyl chloride	75-01-4	0.06	0.06	Not Listed	Not Listed	0.0007	0.005

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

Lab Notes: Reporting Limits and Method Detection Limits are subject to change.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: VOC

Concentration Level: High

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 8260B/EPA 5035A ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Benzene	71-43-2	0.255	0.255	Not Listed	Not Listed	0.004	0.0125
Ethylbenzene	100-41-4	5.16	5.16	Not Listed	Not Listed	0.007	0.0125
Toluene	108-88-3	5.45	5.45	Not Listed	Not Listed	0.00755	0.0125
m&p-Xylene	179601-23-1	3400	3400	Not Listed	Not Listed	0.0148	0.025
o-Xylene	95-47-6	3800	3800	Not Listed	Not Listed	0.00645	0.0125
Xylenes, Total	1330-20-7	10	10	Not Listed	Not Listed	0.00645	0.025
1,2,4-Trimethylbenzene	95-63-6	62	62	Not Listed	Not Listed	0.0149	0.1
1,3,5-Trimethylbenzene	108-67-8	780	780	Not Listed	Not Listed	0.01375	0.1
Bromoform	75-25-2	15.9	15.9	Not Listed	Not Listed	0.02845	0.1
Carbon tetrachloride	56-23-5	0.61	0.61	Not Listed	Not Listed	0.01405	0.05
Chlorobenzene	108-90-7	13.1	13.1	Not Listed	Not Listed	0.0119	0.05
Chloroform	67-66-3	0.29	0.29	Not Listed	Not Listed	0.01245	0.05
1,2-Dichloroethane	107-06-2	0.43	0.43	Not Listed	Not Listed	0.01405	0.05
1,1-Dichloroethene	75-35-4	8.28	8.28	Not Listed	Not Listed	0.0145	0.05
cis-1,2-Dichloroethene	156-59-2	160	160	Not Listed	Not Listed	0.0112	0.05
trans-1,2-Dichloroethene	156-60-5	0.784	0.784	Not Listed	Not Listed	0.01355	0.05
Dichlorobromomethane	75-27-4	0.68	0.68	Not Listed	Not Listed	0.01375	0.1
Dichloromethane	75-09-2	4.05	4.05	Not Listed	Not Listed	0.0315	0.25
1,2-Dichloropropane	78-87-5	0.89	0.89	Not Listed	Not Listed	0.0178	0.05
cis-1,3-Dichloropropene	10061-01-5	0.398	0.398	Not Listed	Not Listed	0.01395	0.05
trans-1,3-Dichloropropene	10061-02-6	0.398	0.398	Not Listed	Not Listed	0.01755	0.05
Styrene	100-42-5	4.69	4.69	Not Listed	Not Listed	0.01305	0.05

Tetrachloroethene	127-18-4	0.55	0.55	Not Listed	Not Listed	0.01085	0.05
1,1,1-Trichloroethane	71-55-6	29.8	29.8	Not Listed	Not Listed	0.0131	0.05
1,1,2-Trichloroethane	79-00-5	1.1	1.1	Not Listed	Not Listed	0.0151	0.05
Trichloroethene	79-01-6	2.8	2.8	Not Listed	Not Listed	0.00755	0.0125
Vinyl chloride	75-01-4	0.06	0.06	Not Listed	Not Listed	0.0063	0.0125

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

Lab Notes: Reporting Limits and Method Detection Limits are subject to change.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: SVOC

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 8270C/EPA 3541A ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
N-Nitrosodiphenylamine	86-30-6	0.545	0.545	Not Listed	Not Listed	0.0449	0.167
N-Nitrosodi-n-propylamine	621-64-7	0.069	0.069	Not Listed	Not Listed	0.0422	0.167
Bis(2-chloroethyl)ether	111-44-4	0.21	0.21	Not Listed	Not Listed	0.0492	0.167
Bis(2-ethylhexyl) phthalate	117-81-7	0.925	0.925	Not Listed	Not Listed	0.044	0.167
1,2-Dichlorobenzene	95-50-1	2.96	2.96	Not Listed	Not Listed	0.0363	0.167
1,4-Dichlorobenzene	106-46-7	0.546	0.546	Not Listed	Not Listed	0.0349	0.167
Hexachlorocyclopentadiene	77-47-4	0.755	0.755	Not Listed	Not Listed	0.154	0.67
1,2,4-Trichlorobenzene	120-82-1	11.1	11.1	Not Listed	Not Listed	0.0376	0.167
Phenol	108-95-2	120	120	Not Listed	Not Listed	0.0526	0.167
2-Methylphenol	95-48-7	40.4	40.4	Not Listed	Not Listed	0.0441	0.167
3 & 4 Methylphenol	15831-10-4	163	163	Not Listed	Not Listed	0.0629	0.167
2,4-Dimethylphenol	105-67-9	0.01*	0.01*	Not Listed	Not Listed	0.104	0.33
Acenaphthene	83-32-9	3400	3400	Not Listed	Not Listed	0.00993	0.033
Acenaphthylene	208-96-8	3400	3400	Not Listed	Not Listed	0.00763	0.033
Anthracene	120-12-7	17,000	17,000	Not Listed	Not Listed	0.00781	0.033
Benzo[a]anthracene	56-55-3	0.15	0.15	Not Listed	Not Listed	0.00696	0.033
Benzo[a]pyrene	50-32-8	0.015	0.015	Not Listed	Not Listed	0.00605	0.033
Benzo[b]fluoranthene	205-99-2	0.15	0.15	Not Listed	Not Listed	0.00645	0.033
Benzo[g,h,i]perylene	191-24-2	1,700	1,700	Not Listed	Not Listed	0.0112	0.033
Benzo[k]fluoranthene	207-08-9	1.5	1.5	Not Listed	Not Listed	0.00792	0.033
Chrysene	218-01-9	15	15	Not Listed	Not Listed	0.0075	0.033
Dibenz(a,h)anthracene	53-70-3	0.015	0.015	Not Listed	Not Listed	0.00928	0.033

Fluoranthene	206-44-0	2300	2300	Not Listed	Not Listed	0.0136	0.033
Fluorene	86-73-7	2300	2300	Not Listed	Not Listed	0.00755	0.033
Indeno[1,2,3-cd]pyrene	193-39-5	0.15	0.15	Not Listed	Not Listed	0.0112	0.033
2-Methylnaphthalene	91-57-6	310	310	Not Listed	Not Listed	0.0431	0.167
Naphthalene	91-20-3	3.6	3.6	Not Listed	Not Listed	0.0064	0.033
Phenanthrene	85-01-8	17,000	17,000	Not Listed	Not Listed	0.0139	0.033
Pyrene	129-00-0	1,700	1,700	Not Listed	Not Listed	0.012	0.033

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

Lab Notes: Reporting Limits and Method Detection Limits are subject to change.

*PAL and PQL are based on the ecological soil risk-based screening levels (SL) for Illinois as presented in the IBS Multi-Site Risk Assessment Framework (Exponent, 2011). If ecological-based SLs are anticipated for the site, a laboratory that can meet the requirements will be identified and utilized.

QAPP Worksheet #15
 (UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica
 Matrix: Soil
 Analytical Group: DRO/ORO
 Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 8015B/EPA 5030B ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Diesel Range Organics (C10-C28)	STL00143	2,000	2,000	Not Listed	Not Listed	3.2	8.3
Oil Range Organics (C28-C40)	STL00293	2,000	2,000	Not Listed	Not Listed	16.7	33.3

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

Lab Notes: Reporting Limits and Method Detection Limits are subject to change.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: GRO

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 8015B/EPA 5030B ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Gasoline Range Organics (C6-C9)	STL00215	2,000	2,000	Not Listed	Not Listed	0.0096	0.05

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

Lab Notes: Reporting Limits and Method Detection Limits are subject to change.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: GRO

Concentration Level: High

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 8015B/EPA 5030B ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Gasoline Range Organics (C6-C9)	STL00215	2,000	2,000	Not Listed	Not Listed	0.379	2.5

¹Analytical MDLs and QLs are those documented in validated methods.²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.**Lab Notes: Reporting Limits and Method Detection Limits are subject to change.**

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: Metals (ICP)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 6010B/EPA 3050B ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Aluminum	7429-90-5	77,000	77,000	Not Listed	Not Listed	1.38	20
Antimony	7440-36-0	0.27	0.27	Not Listed	Not Listed	0.23	2
Arsenic	7440-38-2	0.39	0.39	Not Listed	Not Listed	0.14	1
Barium	7440-39-3	330	330	Not Listed	Not Listed	0.056	1
Cadmium	7440-43-9	0.36	0.36	Not Listed	Not Listed	0.027	0.2
Chromium	7440-47-3	26	26	Not Listed	Not Listed	0.085	1
Copper	7440-50-8	28	28	Not Listed	Not Listed	0.14	1
Iron	7439-89-6	55,000	55,000	Not Listed	Not Listed	2.58	20
Lead	7439-92-1	11	11	Not Listed	Not Listed	0.24	0.5
Manganese	7439-96-5	220	220	Not Listed	Not Listed	0.042	1
Nickel	7440-02-0	38	38	Not Listed	Not Listed	0.066	1
Selenium	7782-49-2	0.52	0.52	Not Listed	Not Listed	0.28	1
Silver	7440-22-4	4.2	4.2	Not Listed	Not Listed	0.063	0.5
Vanadium	7440-62-2	7.8	7.8	Not Listed	Not Listed	0.048	0.5
Zinc	7440-66-6	46	46	Not Listed	Not Listed	0.16	2

¹Analytical MDLs and QLs are those documented in validated methods.²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.**Lab Notes: Reporting Limits and Method Detection Limits are subject to change.**

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: Metals (Hg)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 7471A ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Mercury	7439-97-6	0.1	0.1	Not Listed	Not Listed	0.0051	0.0167

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

Lab Notes: Reporting Limits and Method Detection Limits are subject to change.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Soil

Analytical Group: Cyanide

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit ³ (mg/kg)	EPA 9014B/EPA 9010B ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs (mg/Kg)	QLs (mg/Kg)
Cyanide, Total	57-12-5	1.33	1.33	Not Listed	Not Listed	0.056	0.5

¹Analytical MDLs and QLs are those documented in validated methods.²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.**Lab Notes: Reporting Limits and Method Detection Limits are subject to change.**

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica

Matrix: Air

Analytical Group: VOC

Concentration Level: Low

Analyte	CAS Number	Project Action Limit ⁴		Project Quantitation Limit ³ (ug/m ³)	TO-15 ¹		Achievable Laboratory Limits ²	
		Indoor Air	Shallow Soil Gas		MDLs	Method QLs	MDLs	QLs
		(ug/m ³)	(ug/m ³)				(ug/m ³)	(ug/m ³)
Benzene	71-43-2	0.31	3.1	0.31	Not Listed	Not Listed	0.058	0.65
Toluene	108-88-3	5200	52000	5200	Not Listed	Not Listed	0.069	0.8
Ethylbenzene	100-41-4	0.97	9.7	0.97	Not Listed	Not Listed	0.097	0.88
Xylene (total)	1330-20-7	100	1000	100	Not Listed	Not Listed	0.097	0.88
Naphthalene	91-20-3	0.072	0.72	0.072	Not Listed	Not Listed	0.037	0.068
Chloroform	865-49-6	0.11	1.1	0.11	Not Listed	Not Listed	0.015	0.068
Styrene	100-42-5	1000	10000	1000	Not Listed	Not Listed	0.03	0.2
cis-1,3-Dichloropropene	10061-01-5	0.61	6.1	0.61	Not Listed	Not Listed	0.016	0.2
trans-1,3-Dichloropropene	10061-02-6	0.61	6.1	0.61	Not Listed	Not Listed	0.02	0.2
n-Propylbenzene	103-65-1	1000	10000	1000	Not Listed	Not Listed	0.05	0.2
n-Butylbenzene	104-51-8	---	---	---	Not Listed	Not Listed	0.055	0.2
1,4-Dichlorobenzene	106-46-7	0.22	2.2	0.22	Not Listed	Not Listed	0.044	0.2
1,2-Dibromoethane	106-93-4	0.0041*	0.041	0.0041	Not Listed	Not Listed	0.018	0.2
3-Chloropropene	107-05-1	0.41	4.1	0.41	Not Listed	Not Listed	0.019	0.5
1,2-Dichloroethane	107-06-2	0.094	0.94	0.094	Not Listed	Not Listed	0.031	0.2

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methyl isobutyl ketone	108-10-1	3100	31000	3100	Not Listed	Not Listed	0.026	0.5
1,3,5-Trimethylbenzene	108-67-8	---	---	---	Not Listed	Not Listed	0.051	0.2
Chlorobenzene	108-90-7	52	520	52	Not Listed	Not Listed	0.02	0.2
Tetrahydrofuran	109-99-9	---	---	---	Not Listed	Not Listed	0.018	5
n-Hexane	110-54-3	730	7300	730	Not Listed	Not Listed	0.026	0.2
Cyclohexane	110-82-7	6300	63000	6300	Not Listed	Not Listed	0.039	0.2
1,2,4-Trichlorobenzene	120-82-1	2.1	21	2.1	Not Listed	Not Listed	0.05	0.5
1,4-Dioxane	123-91-1	0.32	---	0.32	Not Listed	Not Listed	0.088	5
Dibromochloromethane	124-48-1	0.09	0.9	0.09	Not Listed	Not Listed	0.021	0.2
Tetrachloroethene	127-18-4	0.41	4.1	0.41	Not Listed	Not Listed	0.011	0.2
sec-Butylbenzene	135-98-8	---	---	---	Not Listed	Not Listed	0.047	0.2
n-Heptane	142-82-5	---	---	---	Not Listed	Not Listed	0.01	0.2
cis-1,2-Dichloroethene	156-59-2	---	---	---	Not Listed	Not Listed	0.014	0.2
trans-1,2-Dichloroethene	156-60-5	63	630	63	Not Listed	Not Listed	0.032	0.2
Methyl tert-butyl ether	1634-04-4	9.4	94	9.4	Not Listed	Not Listed	0.016	0.2
1,2-Dichloroethene, Total	540-59-0	---	---	---	Not Listed	Not Listed	0.014	0.2
1,3-Dichlorobenzene	541-73-1	---	---	---	Not Listed	Not Listed	0.044	0.2
Carbon tetrachloride	56-23-5	0.41	4.1	0.41	Not Listed	Not Listed	0.033	0.2
Methyl Butyl Ketone (2-Hexanone)	591-78-6	31	310	31	Not Listed	Not Listed	0.039	0.5
Isopropyl alcohol	67-63-0	7300	---	7300	Not Listed	Not Listed	0.037	5
Acetone	67-64-1	32000	320000	32000	Not Listed	Not Listed	0.045	5
1,1,1-Trichloroethane	71-55-6	5200	52000	5200	Not Listed	Not Listed	0.035	0.2
Bromomethane	74-83-9	5.2	52	5.2	Not Listed	Not Listed	0.012	0.2
Chloromethane	74-87-3	94	940	94	Not Listed	Not Listed	0.013	0.5
Chloroethane	75-00-3	10000	100000	10000	Not Listed	Not Listed	0.016	0.5
Vinyl chloride	75-01-4	0.16	1.6	0.16	Not Listed	Not Listed	0.029	0.2
Methylene Chloride	75-09-2	5.2	52	5.2	Not Listed	Not Listed	0.013	0.5

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Carbon disulfide	75-15-0	730	7300	730	Not Listed	Not Listed	0.066	0.5
Bromoform	75-25-2	2.2	---	2.2	Not Listed	Not Listed	0.019	0.2
Bromodichloromethane	75-27-4	0.066	0.66	0.066	Not Listed	Not Listed	0.028	0.2
1,1-Dichloroethane	75-34-3	1.5	15	1.5	Not Listed	Not Listed	0.035	0.2
1,1-Dichloroethene	75-35-4	210	2100	210	Not Listed	Not Listed	0.03	0.2
tert-Butyl alcohol	75-65-0	---	---	---	Not Listed	Not Listed	0.071	5
Trichlorofluoromethane	75-69-4	730	7300	730	Not Listed	Not Listed	0.034	0.2
Dichlorodifluoromethane	75-71-8	100	1000	100	Not Listed	Not Listed	0.038	0.5
Freon TF	76-13-1	31000	310000	31000	Not Listed	Not Listed	0.01	0.2
1,2-Dichloropropane	78-87-5	0.24	2.4	0.24	Not Listed	Not Listed	0.014	0.2
Methyl Ethyl Ketone	78-93-3	5200	52000	5200	Not Listed	Not Listed	0.017	0.5
1,1,2-Trichloroethane	79-00-5	0.15	1.5	0.15	Not Listed	Not Listed	0.019	0.2
Trichloroethene	79-01-6	0.43	4.3	0.43	Not Listed	Not Listed	0.03	0.2
1,1,2,2-Tetrachloroethane	79-34-5	0.042	0.42	0.042	Not Listed	Not Listed	0.04	0.2
Methyl methacrylate	80-62-6	730	7300	730	Not Listed	Not Listed	0.013	0.5
Hexachlorobutadiene	87-68-3	0.11	---	0.11	Not Listed	Not Listed	0.065	0.2
2-Chlorotoluene	95-49-8	---	---	---	Not Listed	Not Listed	0.047	0.2
1,2-Dichlorobenzene	95-50-1	210	2100	210	Not Listed	Not Listed	0.048	0.2
1,2,4-Trimethylbenzene	95-63-6	7.3	73	7.3	Not Listed	Not Listed	0.052	0.2
tert-Butylbenzene	98-06-6	---	---	---	Not Listed	Not Listed	0.047	0.2
Cumene	98-82-8	420	4200	420	Not Listed	Not Listed	0.031	0.2
4-Isopropyltoluene	99-87-6	---	---	---	Not Listed	Not Listed	0.048	0.2

¹Analytical MDLs and QLs are those documented in validated methods.²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.⁴Action limit based on screening level for residential, cancer endpoint, CR10⁻⁶. RLs based on 6-liter canister sample.

--- indicates no current action limit for analyte

*MDL exceeds indoor air action limit, however this analyte is not expected to be a driver for IBS sites. Detection limits for analytes will be addressed during the Risk Assessment as necessary

QAPP Worksheet #15
 (UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Laboratory: TestAmerica
 Matrix: Air
 Analytical Group: CO₂, O, N₂, CH₄, Co
 Concentration Level: Low

Analyte	CAS Number	Project Action Limit ⁴	Project Quantitation Limit ³	TO-3 ¹		Achievable Laboratory Limits ²	
				MDLs	Method QLs	MDLs	QLs
		(ppbv)	(ppbv)			(ppbv)	(ppbv)
Carbon Dioxide	124-38-9	---	0.004	Not Listed	Not Listed	0.05	0.004
Oxygen	7782-44-7	---	0.011	Not Listed	Not Listed	0.04	0.011
Methane	74-82-8	---	0.016	Not Listed	Not Listed	0.04	0.016

¹Analytical MDLs and QLs are those documented in validated methods.

²Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

³Site Specific PQLs and RLs are to be established in the Site-Specific Work Plans.

--- indicates no current action limit for analyte

QAPP Worksheet #19

(UFP-QAPP Manual Section 3.1.1)

For each matrix, analytical group, and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference ¹	Sample Volume*	Containers (number, size, and type)**	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Soil	VOC	Low/High	EPA 8260B/EPA 5035A/UP-MV-8260	5 grams each vial	Terracore Kit (4 vials; 2 oz jar)	2-DI, 2-Sodium Bisulfate, 1-MeOH; cool 4 deg C	DI vials 48 hours to freeze; 14 days analysis
Soil	SVOC	Low	EPA 8270C/EPA 3541/UP-MB-8270C/UP-SP-3541	15 grams	4 oz jar; Teflon-lined cap	Cool 4 deg C	14 days/40 days
Soil	DRO/ORO	Low	EPA 8015B/EPA 3541/UP-GE-DRO/UP-SP-3541	15 grams	4 oz jar; Teflon-lined cap	Cool 4 deg C	14 days/40 days
Soil	GRO	Low/High	EPA 8015B/EPA 5030B/5035/UP-GV-GRO	5 grams	2 oz jar; Teflon-lined cap	Cool 4 deg C	14 days/40days
Soil	Metals (ICP)	Low	EPA 6010B/EPA 3050B/UP-ME-6010B/UP-SP-3000	1-2 grams	4 oz jar; Teflon-lined cap	Cool 4 deg C	180 days/180 days
Soil	Metals (Hg)	Low	EPA 7471A	~ 0.5 grams	4 oz jar; Teflon-lined cap	Cool 4 deg C	28 days/28 days
Soil	Cyanide	Low	EPA 9014B/EPA 9010B/UP-WC-CN	1 gram	4 oz jar; Teflon-lined cap	Cool 4 deg C	14 days
Soil	R-Sulfide	Low	EPA 9034/Section 7.3.4/UP-WC-Sulfide	10 grams	4 oz jar; Teflon-lined cap	Cool 4 deg C	7 days
Air	Volatile Organics	Low	TO-15	6 Liters	6 Liter Summa Canister	None	30 Days
Air	Volatile Organics	Low	TO-3c	6 Liters	6 Liter Summa Canister	None	30 Days

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

LAB NOTES:

*Minimal sample volume for analysis

** Some analyses may be combined and collected in one 8 or 16 oz jar

QAPP Worksheet #23

(UFP-QAPP Manual Section 3.2.1)

Analytical SOP References Table

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
UP-MV-8260	Gas Chromatography Mass Spectrometry-Volatiles, 11/20/11, Rev 22	Definitive	VOC	GC/MS	TA Chicago	N
UP-MB-8270C	Gas Chromatography Mass Spectrometry-Semi-Volatiles, SW846 Method EPA 8270C, 11/18/11, Rev 20	Definitive	SVOC	GC/MS	TA Chicago	N
UP-GE-DRO	Gas Chromatography: Semi-Volatiles Diesel Range Organics (DRO), 09/30/2011, Rev 15	Definitive	DRO/ORO	GC	TA Chicago	N
UP-SP-3541	Sample Preparation Semivolatile and Nonvolatile Organic Compounds from a Soil/Sediment Matrix using Soxhlet Extraction, 10/03/2011, Rev 10	Definitive	Sample Preparation (SVOC, DRO/ORO)	NA	TA Chicago	N
UP-GV-GRO	Gas Chromatography: Volatiles Gasoline Range Organics (GRO), 09/30/2011, Rev 15	Definitive	GRO	GC	TA Chicago	N
UP-ME-6010B	Metals Analysis Trace Inductively Coupled Argon Plasma by SW846 6010B (Simultaneous Operation), 09/30/2011, Rev 14	Definitive	Metals (ICP)	Trace ICP	TA Chicago	N
UP-SP-3000	Sample Preparation Metals Digestion by SW-846 3000 Series, 03/03/2011, Rev 20	Definitive	Sample Preparation (Metals ICP)	NA	TA Chicago	N
UP-ME-245.1	Metals Analysis Mercury by EPA Methods 245.1/245.5; SW-846 7470A/7471A/7471B; and U.S. EPA CLP Doc No ILM04.0, 10/28/11, Rev 17	Definitive	Metals (Hg)	Mercury Analyzer	TA Chicago	N
UP-WC-CN	Wet Chemistry Cyanide (Total/Weak Acid Dissociable/Amenable/Reactive), 10/28/2011, Rev 23	Definitive	Cyanide	UV Spec	TA Chicago	N
UP-WC-Sulfide	Wet Chemistry Total Acid Soluble, Acid-Volatile and Reactive Sulfide, 11/01/2010, Rev 15	Definitive	Sulfide	Titration	TA Chicago	N

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A-1	Determination of VOCs in Ambient Air (EPA Compendium Method TO15), BR-AT-004, 09/25/09, Rev 7	Definitive	VOC	GC/MS	TestAmerica, Burlington	N
LA-MSA-151	Determination of Low-Level VOCs in Ambient/Indoor Whole Air Samples using GC/MS-SIM Mode LA-MSA-151, Rev 6	Definitive	VOC	GC/MS	TestAmerica, Los Angeles	N
BR-AT-002	Determination of Carbon Dioxide, Oxygen, Nitrogen, Methane, and Carbon Monoxide by GC/TCD BR-AT-002, Rev 2	Definitive	CO ₂ , O, N ₂ , CH ₄ , Co	GC/TCD	TestAmerica, Burlington	N

QAPP Worksheet #24

(UFP-QAPP Manual Section 3.2.2)

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference¹
GC/MS	Tune (BFB, DFTPP)	Prior to initial calibration or continuing calibration; every 12 hours	Refer to SOP	Correct problem; re-analyze tune	Analyst	UP-MV-8260; UP-MB-8270C
GC/MS	Initial Calibration	Prior to sample analysis or as needed	VOC : CCC \leq 30% RSD; SPCC \geq 0.300 or 0.100; all other targets < 15% RSD; linear $r \geq$ 0.995 SVOC: CCC \leq 30% RSD; SPCC \geq 0.05; ; all other targets grand mean < 15% RSD; linear $r \geq$ 0.995	Correct problem; repeat initial calibration	Analyst	UP-MV-8260; UP-MB-8270C
GC/MS	Continuing Calibration	Daily, before sample analysis and every 12 hours of tune time	VOC : CCC \leq 20% DIFF/Drift SPCC \geq 0.300 or 0.100; SVOC: CCC \leq 20% DIFF/Drift SPCC \geq 0.05	Correct problem and repeat CCV and associated samples; repeat initial calibration if necessary and CCV and samples; may report non-detects if biased high.	Analyst	UP-MV-8260; UP-MB-8270C
GC	Initial Calibration	Prior to sample analysis or as needed	CF RSD \leq 20%; or linear $r \geq$ 0.995	Correct problem; repeat initial calibration	Analyst	UP-GE-DRO; UP-GE-GRO
GC	Continuing Calibration	Before sample analysis, every 10 samples and end of sequence	All analytes within 15% of expected value and within RT window. Average of all analytes 15%.	Repeat CCV once; correct problem; re-analyze initial calibration, CCV and all bracketed samples; may report non-detects if biased high	Analyst	UP-GE-DRO; UP-GE-GRO

Metals (ICP)	Initial Calibration	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem; repeat initial calibration	Analyst	UP-ME-6010B
Metals (ICP)	Continuing Calibration	After every 10 readings and end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements and bracketed samples; may report non-detects if biased high	Analyst	UP-ME-6010B
Metals (Hg)	Initial Calibration	Quarterly, monthly or daily-see SOP	$r \geq 0.995$	Correct problem; repeat initial calibration	Analyst	UP-ME-245.1
Metals (Hg)	Continuing Calibration	Beginning, every 10 samples and at end of sequence	+/- 10% of known concentration	Correct problem then repeat CCV, CCB and bracketed samples; may report non-detects if biased high	Analyst	UP-ME-245.1
Cyanide	Initial Calibration	Daily initial calibration prior to sample analysis	$r \geq 0.995$	Correct problem; repeat initial calibration	Analyst	UP-WC-CN
Cyanide	Continuing Calibration	After every 10 readings and at end of sequence	+/- 10% of known concentration	Correct problem then repeat CCV, CCB and bracketed samples; may report non-detects if biased high	Analyst	UP-WC-CN
GC/MS	Initial Calibration	Prior to sample analysis and when CCV fails	RSD for each analyte $\leq 30\%$ with 2 exceptions up to 40%	Correct problem and repeat calibration	Analyst	A-1
GC/MS	ICV	Once after each ICAL	%R for all analytes within 70-130	Correct Problem. Reanalyze, re-make, re-verify & re-analyze. If that fails, re-make all standards and repeat calibration.	Analyst	A-1
GC/MS	CCV	Daily before sample analysis after tune standard	%D ≤ 30	Correct Problem. Reanalyze once. If that fails, see section 10.2.5 for instruction.	Analyst	A-1

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

Lab Notes: See LQSM and Analytical Standard Operating Procedures (SOPs) for full details.

QAPP Worksheet #25

(UFP-QAPP Manual Section 3.2.3)

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference¹
Hewlett Packard GC/MS (Air)	Routine	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	As required	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

Lab Note: All maintenance procedures will follow the Laboratory Quality System Manual and Analytical Standard Operating Procedures (SOPs). The LQSM and SOPs are available upon request.

QAPP Worksheet #30

(UFP-QAPP Manual Section 3.5.2.3)

Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Soil	VOC	Low	Amendment, Overburden, Excavation	UP-MV-8260	10 business days	TestAmerica Chicago, 2417 Bond Street, University Park, IL 60484, Marilyn Krueding, (708) 534-5200	
Soil	SVOC	Low	Amendment, Overburden, Excavation	UP-MB-8270C	10 business days	TestAmerica Chicago, 2417 Bond Street, University Park, IL 60484, Marilyn Krueding, (708) 534-5200	
Soil	DRO/ORO	Low	Amendment, Overburden, Excavation	UP-GE-DRO,	10 business days	TestAmerica Chicago, 2417 Bond Street, University Park, IL 60484, Marilyn Krueding, (708) 534-5200	
Soil	GRO	Low	Amendment, Overburden, Excavation	UP-GV-GRO,	10 business days	TestAmerica Chicago, 2417 Bond Street, University Park, IL 60484, Marilyn Krueding, (708) 534-5200	
Soil	Metals	Low	Amendment, Overburden, Excavation	UP-ME-6010B, UP-ME-245.1	10 business days	TestAmerica Chicago, 2417 Bond Street, University Park, IL 60484, Marilyn Krueding, (708) 534-5200	
Soil	Cyanide	Low	Amendment, Overburden, Excavation	UP-WC-CN	10 business days	TestAmerica Chicago, 2417 Bond Street, University Park, IL 60484, Marilyn Krueding, (708) 534-5200	
Air	VOCs	Low	Air Monitoring	A-1	10 business days	TestAmerica Burlington 30 Community Drive, Suite 11 South Burlington, VT 05403	
Air	VOCs	Indoor Air	Air Monitoring	LA-MSA-151	10 business days	TestAmerica Los Angeles 3585 Cadillac Avenue, Suite A Costa Mesa, CA 92626	
Air	CO ₂ , O, N ₂ , CH ₄ , Co	Low	Air Monitoring	BR-AT-002	10 business days	TestAmerica Burlington 30 Community Drive, Suite 11 South Burlington, VT 05403	



Enclosure D

**Addendum No. 1, Revision
1, to Site-Specific Work
Plan, Revision 1 for the
Hough Place Station
Former MGP Site (NRT,
August 29, 2014)**



ENVIRONMENTAL CONSULTANTS

300 S. WACKER DRIVE, SUITE 2050
CHICAGO, ILLINOIS 60606
(P) 312.465.1740
(F) 414.837.3608

Mr. Ross del Rosario
USEPA Region 5 – SR-6J
77 W. Jackson Boulevard
Chicago, Illinois 60604-3590

August 29, 2014
(2116)

RE: Addendum No. 1, Revision 1 to Site-Specific Work Plan, Revision 1
Modified Sampling and Analysis Protocols for Slow-Recharging Wells
Hough Place Station Former MGP, Upland OU, South Branch Site, Chicago, Illinois
The Peoples Gas Light and Coke Company
CERCLA Docket No. V-W-08-C-917
CERCLIS ID – ILN000510190

Dear Mr. del Rosario:

On behalf of Integrys Business Support, LLC (IBS), Natural Resource Technology, Inc. (NRT) is providing the enclosed one hard copy and CD copies of this Addendum No. 1, Revision 1 to the Site Specific Work Plan Revision 1 (SSWP Rev 1) of the Hough Place Station Site Former Manufactured Gas Plant (MGP), dated July 17, 2013. Updates to the SSWP Rev 1 have been prepared consistent with Section 1.1.2.2 of the Statement of Work (SOW) included with the Settlement Agreement and Administrative Order on Consent (Settlement Agreement) between United States Environmental Protection Agency (USEPA) and The Peoples Gas Light and Coke Company (PGL) effective October 31, 2008. Specifically, this letter seeks approval from USEPA to modify sampling and analysis protocols to address site-specific conditions encountered during the Remedial Investigation (RI) activities at the Hough Place Station Site (initiated in May 2014).

Following the criteria outlined in the Well Nest Installation Decision Tree (Table H4 of the SSWP Rev1), piezometers were typically installed by drilling approximately 17 feet into the native clay and inserting a 10-foot well screen. Field observations recorded during the well development activities indicate that the piezometers exhibit slow recharge rates due to the low permeability of the native clay. In some cases during quarterly sampling events, piezometers may not produce a sufficient volume of water within 24 hours of purging to fill sample bottles required for analysis of all groundwater COPCs described in the SSWP, Rev 1. These conditions have also been observed in some piezometers recently installed at Throop Street Station, Pitney Court Station and Crawford Station Former MGP Sites. To develop additional options for sampling slow-recharging wells, well purge and sampling techniques and guidance were reviewed.

Recommendations to modify sampling and analysis protocols to address site-specific conditions and slow-recharging wells are detailed below.

Well Purge Considerations

A description of well purging procedures included in paragraph 1 of Section 3.5.8 of the Hough Place Station SSWP Rev 1, stated the following:

"All network wells will be sampled within a reasonably short time. Low-flow groundwater sampling will be completed for all wells using a peristaltic pump as described in USEPA-approved SOP SAS-08-02 and Section 4 of the Multi-Site FSP, provided the depth to water after sampling does not exceed 15 feet. If water depths exceed 15 feet after sampling, regardless of stability, subsequent low-flow sampling events will be conducted using a submersible pump. In the event that a well does not produce sufficient water for using these methods (i.e., water column less than 2 feet or very low recharge rates), the well will be purged dry, allowed to recharge and sampled with a bailer within approximately 24 hours of purging. This procedure is consistent with USEPA-approved SOP SAS-08-03."

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Identifying additional well purging options for the many slow-recharging wells at these sites is desirable. Guidance from the most current, comprehensive USEPA groundwater sampling document (USEPA Region 4, *Groundwater Sampling, SESDPROC-301-R3* [2013]) with respect to well purging guidance (Attachment A) was reviewed. Guidance from this document applicable to slow-recharging wells is excerpted below.

3.2.1.1.3 Purge Adequacy Considerations

"In some situations, even with slow purge rates, a well may be pumped or bailed dry (evacuated). In these situations, this generally constitutes an adequate purge and the well can be sampled following sufficient recovery (enough volume to allow filling of all sample containers). It is not necessary that the well be evacuated three times before it is sampled. The pH, specific conductance, temperature, and turbidity should be measured and recorded, during collection of the sample from the recovered volume, as the measurements of record for the sampling event. For wells with slow recovery, attempts should be made to avoid purging them to dryness. This can be accomplished, for example, by slowing the purge rate.

"It is particularly important that wells be sampled as soon as possible after purging. If adequate volume is available immediately upon completion of purging, the well must be sampled immediately. If not, sampling should occur as soon as adequate volume has recovered. If possible, sampling of wells which have a slow recovery should be scheduled so that they can be purged and sampled in the same day, after adequate volume has recovered. Wells of this type should, unless it is unavoidable, not be purged at the end of one day and sampled the following day."

Based on this USEPA guidance, a slow-recharging well is defined herein as a well that can be purged dry during low-flow groundwater sampling (per USEPA-approved SOP SAS-08-02), prior to the measured stabilization of field parameters.

Sample Volume Considerations

Identifying appropriate modifications to sample volume requirements as options for slow-recharging wells is also desirable. Analytical laboratories were contacted to determine the minimum sample volumes required to perform the analyses set forth in the Hough Place Station SSWP Rev 1. Based on the lab responses, sample volumes can be significantly reduced, if necessary:

- Polynuclear aromatic hydrocarbons (PAH) in water by EPA Methods 3510/8270 SIM require only 100 mL of sample (Attachment B). The sample volume required for the original 8270 sample preparation was 1 L. The smaller volume prep and analysis methods 3510/8270 SIM have become an industry standard.
- Metals by EPA Method 6010/6020 requires a minimum of 60 mL compared to the standard volume of 250 mL
- Volatile organic compounds (VOC) by EPA Method 8260 requires one 40-mL vial compared to the standard volume of 3, 40-mL vials

The PAH method referenced above allows the lab to run all necessary investigative and quality assurance testing on a smaller volume sample. In the cases of the metals and VOC methods presented above, reducing sample volumes limits the lab's opportunity for sample re-analysis and using those samples for quality assurance (i.e. lab duplicates, lab control samples, etc.). While the larger sample volumes are desirable for these methods because they offer more options for the labs, they are not always achievable.

Recommendations

Based on the above discussion, IBS proposes the following approach to purging and sampling slow-recharging wells at the Hough Place Station Site and other sites within the Multi-Site Program:

- To reduce sample volumes, take advantage of an industry standard, and maintain consistency in groundwater analytical methods throughout the Multi-Site Program, PAHs will be analyzed by the low-volume (aka high-volume injection) EPA Methods 3510/8270 SIM in all monitoring wells, whether they are slow-recharging wells or not. These methods require only 100 mL of sample, whereas the volume required for the original 8270 sample preparation was 1 L. Pace Analytical Services, Inc. (Pace), or TestAmerica labs will be used for analysis of PAHs unless or until approval is obtained to use additional labs for this analysis. Both Pace and TestAmerica labs are approved for use in the Multi-Site Program as documented in the Multi-Site QAPP, Revision 2 (NRT, 2008). Additional backup information for both labs regarding use of EPA Methods 3510/8270 SIM is included in Attachments B and C.
- Slow-recharging wells (i.e. wells that are fully dewatered during purging before field parameters stabilize) will be gauged until a sufficient volume of water is present to collect the required standard volumes for sample analyses. The maximum period of time between when the well is initially purged dry and sample collection will not exceed 24 hours.
- In the event that a well is fully dewatered during purging and gauging the well indicates that an insufficient volume of water is/will be present in the well to collect the required standard volumes for sample analyses within 24 hours, the sample volumes for VOCs and metals may be reduced as follows:

Analytes	Method	Minimum Sample Volume
VOCs	EPA Method 8260	One 40 mL vial
Metals	EPA Method 6010/6020	60 mL

If insufficient sample volume is available to perform all required analysis, sample collection will be prioritized in the following order: VOCs, PAHs, then metals. If less than 40 mL is present in a well after 24 hours, no samples will be collected. Use of reduced sample volumes and/or prioritized sample collection will be documented in field notes. In addition, any deviation to the purging or sampling protocol included herein will be documented in the field notes.

Summary

IBS seeks approval of this Addendum No. 1, Revision 1 to the Hough Place Station SSWP Rev 1, Modified Sampling and Analysis Protocols for Slow-Recharging Wells, for use during future groundwater sampling events. If approved, this protocol will also be applied at other sites within the Multi-Site Program that have slow-recharging wells and use of the low-volume method for PAHs in water will be added to the Multi-Site QAPP. Please contact Mr. Naren Prasad of IBS at 312.240.4569 if you should have any questions regarding the content of this letter.

Sincerely,

NATURAL RESOURCE TECHNOLOGY, INC.



Mark Castro
Scientist/Project Manager



Jennifer M. Hagen, PE
Senior Engineer

Enc: Attachment A USEPA -Region 4 *Groundwater Sampling, SESDPROC-301-R3* (2013)
Attachment B
Attachment B1 Pace Analytical EPA Method 3510C SOP
Attachment B2 Pace Analytical EPA Method 8270C SIM SOP
Attachment B3 UFP QAPP Worksheet 15 for Pace
Attachment B4 UFP QAPP Worksheets 24 and 25 for Pace
Attachment C
Attachment C1 TestAmerica Analytical EPA Method 3510C SOP
Attachment C2 TestAmerica EPA Method 8270C SOP
Attachment C3 UFP QAPP Worksheet 15 for TestAmerica
Attachment C4 UFP QAPP Worksheets 24 and 25 for TestAmerica

cc: Mr. D. Wilson, IEPA (via US Mail)
Mr. Naren Prasad, IBS (via email)
Mr. David Klatt, CH2MHill (via email)

[File:\Hough SSWP Rev 1 Addendum 1 Well Sampling Letter Rev1_140829]

ATTACHMENT A

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Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: Groundwater Sampling

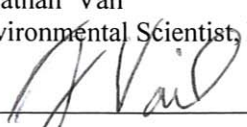
Effective Date: March 6, 2013

Number: SESDPROC-301-R3

Authors

Name: Jonathan Vail

Title: Environmental Scientist, Regional Expert

Signature: 

Date: 3/4/2013

Approvals

Name: Danny France


Title: Chief, Enforcement and Investigations Branch

Signature: 

Date: 3/4/13

Name: Bobby Lewis

Title: Field Quality Manager, Science and Ecosystem Support Division

Signature: 

Date: 3/4/13

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-301-R3, Groundwater Sampling, replaces SESDPROC-301-R2. General: Corrected any typographical, grammatical and/or editorial errors. Title Page: Changed author from Donald Hunter to Jonathan Vail. Changed Enforcement and Investigations Branch Chief from Archie Lee to Danny France. Revision History: Changes were made to reflect the current practice of only including the most recent changes in the revision history. Section 2.3: Item 4 was revised to reflect practice of using individual single-use preservative vials instead of preservatives prepared by ASB.	March 6, 2013
SESDPROC-301-R2, Groundwater Sampling, replaces SESDPROC-301-R1.	October 28, 2011
SESDPROC-301-R1, Groundwater Sampling, replaces SESDPROC-301-R0.	November 1, 2007
SESDPROC-301-R0, Groundwater Sampling, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting groundwater samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling groundwater samples in the field. On the occasion that SESD field personnel determine that any of the procedures described are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a groundwater sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Columbia Analytical Services, Lab Science News, Passive Diffusion Devices & Polyethylene Diffusion Bag (PDB) Samplers.

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

Puls, Robert W., and Michael J. Barcelona. 1989. Filtration of Ground Water Samples for Metals Analysis. Hazardous Waste and Hazardous Materials 6(4), pp.385-393.

Puls, Robert W., Don A. Clark, and Bert Bledsoe. 1992. Metals in Ground Water: Sampling Artifacts and Reproducibility. Hazardous Waste and Hazardous Materials 9(2), pp. 149-162.

SESD Guidance Document, Design and Installation of Monitoring Wells, SESDGUID-001, Most Recent Version

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SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Temperature Measurement, SESDPROC-102, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Groundwater Level and Well Depth Measurement, SESDPROC-105, Most Recent Version

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

SESD Operating Procedure for Pump Operation, SESDPROC-203, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Potable Water Supply Sampling, SESDPROC-305, Most Recent Version

The Interstate Technology & Regulatory Council, Technology Overview of Passive Sampler Technologies, Prepared by The Interstate Technology & Regulatory Council Diffusion Sampler Team, March 2006.

United States Environmental Protection Agency (US EPA). 1975. Handbook for Evaluating Water Bacteriological Laboratories. Office of Research and Development (ORD), Municipal Environmental Research Laboratory, Cincinnati, Ohio.

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US EPA. 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 1995. Ground Water Sampling - A Workshop Summary. Proceedings from the Dallas, Texas November 30 – December 2, 1993 Workshop. ORD, Robert S. Kerr Environmental Research Laboratory. EPA/600/R-94/205, January 1995.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SEDS, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SEDS, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting groundwater samples. Refer to the SEDS Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting groundwater samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Always sample from the anticipated cleanest, i.e., least contaminated location, to the most contaminated location. This minimizes the opportunity for cross-contamination to occur during sampling.
- Collected samples must remain in the custody of the sampler or sample custodian until the samples are relinquished to another party.

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- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and placed in the project files.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Groundwater samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas unpreserved samples have only a seven-day holding time. In the great majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the groundwater has a high amount of dissolved limestone, i.e., is highly calcareous, there will most likely be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment, such as:

- Bailers must be constructed of stainless steel or Teflon®
- RediFlo2® submersible pumps used for sampling should be equipped with Teflon® sample delivery tubing
- Peristaltic pump/vacuum jug assemblies should be outfitted with Teflon® tubing from the water column to the transfer cap, which should also be constructed of Teflon®

2.2 Special Precautions for Trace Contaminant Groundwater Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.

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- Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Clean plastic sheeting will be placed on the ground at each sample location to prevent or minimize contaminating sampling equipment by accidental contact with the ground surface.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Groundwater samples will typically be collected from the discharge line of a pump or from a bailer, either from the pour stream of an up-turned bailer or from the stream from a bottom-emptying device. Efforts should be made to reduce the flow from either the pump discharge line or the bailer during sample collection to minimize sample agitation.
2. During sample collection, make sure that the pump discharge line or the bailer does not contact the sample container.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC, acidity and alkalinity analysis must not have any headspace. All other sample containers must be filled with an allowance for ullage.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by ASB personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples except for the samples collected for VOC analysis. If additional preservative is needed, it should be added to achieve adequate preservation. Preservation requirements for groundwater samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM).

2.4 Quality Control

If possible, a control sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same water-bearing formation. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Procedure for Sample and Evidence Management, SESDPROC-005.

3 Groundwater Sampling Methods – Purging

3.1 General

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of actual aquifer conditions. In order to determine when a well has been adequately purged, field investigators should monitor, at a minimum, the pH, specific conductance and turbidity of the groundwater removed during purging and, in the case of permanent monitoring wells, observe and record the volume of water removed.

There are several purging strategies that may be used, depending on specific conditions encountered for given well sampling situations. When a specific well is characterized, based on the field investigators experience and knowledge, as having fairly typical water levels, depths and purge volumes, as determined according to the procedures in Section 3.2.1, below, SESD will normally use the multiple volume purging procedures and equipment described in Sections 3.2.1 and 3.3 of this procedure for purging the well.

When the traditional multiple volume purge method is considered and it is determined that excessive quantities of IDW would be generated using this method, it may be appropriate, under very limited and specific circumstances, to use an alternate method that reduces the time and amount of purge water to be removed prior to sampling the well. The field project leader will select the alternate method only after careful consideration of the conditions presented by the well and the impact these conditions have on all aspects of the sampling event (time required to sample, quantities of IDW requiring management, etc.).

The alternate purge procedures or sampling strategies available are the “Tubing-in-Screened Interval” method and the MicroPurge or No-Purge methods. These are described and discussed in Sections 3.2.2 and 4.5 of this operating procedure, respectively.

3.2 Purging Methods and Strategies

3.2.1 Traditional Multiple Volume Purge

3.2.1.1 Purging and Purge Adequacy

3.2.1.1.1 Purge Volume Determination

Prior to initiating the purge, the amount of water standing in the water column (water inside the well riser and screen) should be determined, if possible. To do this, the diameter of the well should be determined and the water level and total depth of the well should be measured and recorded. Specific methodology for obtaining these measurements is found in SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105).

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Once this information is obtained, the volume of water to be purged can be determined using one of several methods. One is the equation:

$$V = 0.041 d^2 h$$

Where: h = depth of water in feet

d = diameter of well in inches

V = volume of water in gallons

Alternatively, the volume of standing water in the well and the volume of three water columns may be determined using a casing volume per foot factor for the appropriate diameter well, similar to that in Table 3.2.1. The water level is subtracted from the total depth, providing the length of the water column. This length is multiplied by the appropriate factor in the Table 3.2.1, corresponding to either the single well volume or the triple well volume, to determine both the single well volume and triple well volumes, in gallons, for the well in question. Other acceptable methods include the use of nomographs or other equations or formulae.

TABLE 3.2.1: WELL CASING DIAMETER VOLUME FACTORS

Casing Diameter (inches)	Gallons/ft, One Water Column	Gallons/ft, Three Water Columns
1	0.04	0.12
2	0.16	0.48
3	0.37	1.11
4	0.65	1.98
5	1.02	3.06
6	1.47	4.41
7	1.99	5.97
8	2.61	7.83
9	3.30	9.90
10	4.08	12.24
11	4.93	14.79
12	5.87	17.61

With respect to volume, an adequate purge is normally achieved when three to five well volumes have been removed. The field notes should reflect the single well volume calculations or determinations, according to one of the above methods, and a reference to the appropriate

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multiplication of that volume, i.e., a minimum three well volumes, clearly identified as a purge volume goal.

3.2.1.1.2 Chemical Parameter Stabilization Criteria

With respect to the ground water chemistry, an adequate purge is achieved when the pH and specific conductance of the ground water have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs) (twice the Primary Drinking Water Standard of 5 NTUs). Although 10 NTUs is normally considered the minimum goal for most ground water sampling objectives, lower turbidity has been shown to be easily achievable in most situations and reasonable attempts should be made to achieve these lower levels. (Note: Because groundwater temperature is subject to rapid changes when collected for parameter measurement, its usefulness is subject to question for the purpose of determining parameter stability. As such, it has been removed from the list of parameters used for stability determination. Even though temperature is not used to determine stability during well purging, it is still advisable to record the sample temperature, along with the other groundwater chemistry parameters during well purging, as it may be needed to interpret other chemical parameter results in some situations.)

Stabilization occurs when, for at least three consecutive measurements, the pH remains constant within 0.1 Standard Unit (SU) and specific conductance varies no more than approximately 5 percent. Other parameters, such as dissolved oxygen (DO), may also be used as a purge adequacy parameter. Normal goals for DO are 0.2 mg/L or 10% saturation, whichever is greater. DO measurements must be conducted using either a flow-through cell or an over-topping cell to minimize or reduce any oxygenation of the sample during measurement. Oxidation Reduction Potential (ORP) should not be used as a purge stabilization parameter but may be measured during purging to obtain the measurement of record for ORP for the sampling event.

There are no set criteria for establishing how many total sets of measurements are adequate to document stability of parameters. If the calculated purge volume is small, the measurements should be taken frequently enough to provide a sufficient number of measurements to evaluate stability. If the purge volume is large, measurements taken every 15 minutes, for example, may be sufficient. See the SESD Operating Procedures for Field pH Measurement (SESDPROC-100), Field Specific Conductance Measurement (SESDPROC-101), Field Temperature Measurement (SESDPROC-102), Field Turbidity Measurement (SESDPROC-103), Field Measurement of Dissolved Oxygen (SESDPROC-106) and Field Measurement of Oxidation-Reduction Potential (SESDPROC-113) for procedures for conducting these measurements.

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If, after three well volumes have been removed, the chemical parameters have not stabilized according to the above criteria, additional well volumes (up to five well volumes), should be removed. If the parameters have not stabilized within five volumes, it is at the discretion of the project leader whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible, with respect to turbidity. The conditions of sampling should be noted in the field log.

3.2.1.1.3 Purge Adequacy Considerations

In some situations, even with slow purge rates, a well may be pumped or bailed dry (evacuated). In these situations, this generally constitutes an adequate purge and the well can be sampled following sufficient recovery (enough volume to allow filling of all sample containers). ***It is not necessary that the well be evacuated three times before it is sampled.*** The pH, specific conductance, temperature, and turbidity should be measured and recorded, during collection of the sample from the recovered volume, as the measurements of record for the sampling event.

For wells with slow recovery, attempts should be made to avoid purging them to dryness. This can be accomplished, for example, by slowing the purge rate. As water enters a well that has been purged to dryness, it may cascade down the sand pack and/or the well screen, stripping volatile organic constituents that may be present and/or introducing soil fines into the water column.

It is particularly important that wells be sampled as soon as possible after purging. If adequate volume is available immediately upon completion of purging, the well must be sampled immediately. If not, sampling should occur as soon as adequate volume has recovered. If possible, sampling of wells which have a slow recovery should be scheduled so that they can be purged and sampled in the same day, after adequate volume has recovered. Wells of this type should, unless it is unavoidable, not be purged at the end of one day and sampled the following day.

3.2.2 “Tubing-in-Screened-Interval” Method

The “Tubing-in-Screen” method, sometimes referred to as the “Low Flow” method, is used primarily when calculated purge volumes for the traditional purging method are excessive and present issues related to timely completion of the project and/or management of investigation derived waste.

3.2.2.1 Purge Criteria

3.2.2.1.1 Placement of Pump Tubing or Intake

The peristaltic pump tubing or intake point of the submersible pump is placed in the approximate mid-portion of the screened interval of the well. By definition, this method cannot be applied for purging with a bailer.

3.2.2.1.2 Conditions of Pumping

Prior to initiation of pumping, a properly decontaminated well sounder should be lowered into the well being sampled to monitor the static water level prior to and during the purging process. Ideally, there should be only a slight and stable drawdown of the water column after pumping begins. If this condition cannot be met, then one of the other methods should be employed.

3.2.2.1.3 Stability of Chemical Parameters

As with the traditional purging method described in Section 3.2.1, it is important that all chemical parameters be stable as defined in Section 3.2.1.1 prior to sampling.

3.3 Equipment Considerations for Purging

Monitoring well purging is accomplished by using in-place plumbing and dedicated pumps or by using portable pumps/equipment when dedicated systems are not present. The equipment utilized by Branch personnel will usually consist of peristaltic pumps and variable speed electric submersible pumps, but may also include bladder pumps or inertial pumps. The pump of choice is usually a function of the well diameter, the depth to water, the depth of the well and the amount of water that is to be removed during purging. Whenever the head difference between the sampling location and the water level is less than the limit of suction and the volume to be removed is reasonably small, a peristaltic pump should be used for purging. For wells where the water level is below the limit of suction (approximately 25' to 30', and/or where there is a large volume of water to be purged), the variable speed electric submersible pump would be the pump of choice. SESD Operating Procedure for Pump Operation (SESDPROC-203) contains the use and operating instructions for all pumps commonly used during SESD ground water investigations.

Bailers may also be used for purging in appropriate situations, however, their use is discouraged. Bailers tend to disturb any sediment that may be present in the well, creating or increasing sample turbidity. Bailers, if improperly used, may also strip volatile organic compounds from the water column being sampled. If a bailer is used, it should be a closed-top Teflon® bailer.

3.3.1 Wells Without Plumbing or In-Place Pumps

For permanent monitoring wells, the depth to water (water level) and depth of the well (total depth) should be determined before purging. Caution should be exercised during this procedure to prevent cross-contamination between wells. This is a critical concern when samples for trace organic compounds or metals analyses are collected. See SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) for cleaning procedures for well sounders. After cleaning, the well sounding device should be protected to keep it clean until its next use.

3.3.1.1 Purging with Pumps

3.3.1.1.1 Peristaltic Pumps

The following step-by-step procedures describe the process of purging with a peristaltic pump:

1. Cut a length of standard-cleaned (SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206)) Teflon® tubing, equal to the well depth plus an additional five to ten feet. Enough tubing is needed to run from the ground surface up to the top of the well casing and back down to the bottom of the well. This will allow for operation of the pump at all possible water level conditions in the well.
2. Place one end of the tubing into the vacuum side of the peristaltic pump head. Proper sizing of the Teflon® and Silastic® or Tygon® tubing should allow for a snug fit of the Teflon® tubing inside the flexible tubing mounted in the pump head.
3. Run a short section of tubing (does not have to be Teflon®) from the discharge side of the pump head to a graduated bucket.
4. Place the free end of the Teflon® tubing into the well until the end of the tubing is just below the surface of the water column.
5. Secure the Teflon® tubing to the well casing or other secure object using electrician's tape or other suitable means. This will prevent the tubing from being lost in the well should the tubing detach from the pump head.
6. Turn on the pump to produce a vacuum on the well side of the pump head and begin the purge. Observe pump direction to ensure that a vacuum is being applied to the purge line. If the purge line is being pressurized, either switch the tubing at the pump head or reverse the polarity of the cables on the pump or on the battery.

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7. If the pumping rate exceeds the recovery rate of the well, continue to lower the tubing into the well, as needed, until the drawdown stabilizes or the well is evacuated to dryness. If the pump is a variable speed peristaltic pump, and the water level in the well is being drawn down, reduce the speed of the pump in an attempt to stabilize the drawdown. If the well can be purged without evacuating the well to dryness, a sample with greater integrity can be obtained.
8. For wells which are not evacuated to dryness, particularly those with recovery rates equal to or very nearly equal to the purge rate, there may not be a complete exchange and removal of stagnant water in that portion of the water column above the tubing intake. For this reason, it is important that the tubing intake be placed in the very uppermost portion of the water column while purging. Standard field measurements should frequently be taken during this process to verify adequacy of the purge and readiness for sampling, as described in Section 3.

3.3.1.1.2 Submersible Pumps

When a submersible pump is used for well purging, the pump itself is lowered into the water column. The pump must be cleaned as specified in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

The pump/hose assembly used in purging should be lowered into the top of the standing water column and not deep into the column. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump is placed deep into the water column, the water above the pump may not be removed, and the subsequent samples, particularly if collected with a bailer, may not be representative of the aquifer conditions. It is recommended that the pump not be lowered more than three to five feet into the water column. If the recovery rate of the well is faster than the pump rate and no observable draw down occurs, the pump should be raised until the intake is within one foot of the top of the water column for the duration of purging. If the pump rate exceeds the recovery rate of the well, the pump will have to be lowered, as needed, to accommodate the drawdown. After the pump is removed from the well, the hose and the pump should be cleaned as outlined in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

3.3.1.2 Purging with Bailers

Standard-cleaned (SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SEDS Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206)), closed top Teflon® bailers with Teflon® coated stainless steel leaders and new nylon rope are lowered into the top of the water column, allowed to fill, and removed. It is critical that bailers be slowly and gently immersed into the top of the water column, particularly during final stages of purging, to minimize turbidity and disturbance of volatile organic constituents. The use of bailers for purging and sampling is discouraged because the correct technique is highly operator dependent and improper use may result in an unrepresentative sample.

3.3.2 Wells With In-Place Plumbing

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Many permanent monitoring wells at active facilities are also equipped with dedicated, in-place pumps. The objective of purging wells with in-place pumps is the same as with monitoring wells without in-place pumps, i.e., to ultimately collect a ground water sample representative of aquifer conditions. Among the types of wells identified in this section, two different approaches are necessary.

A permanent monitoring well with an in-place pump should, in all respects, be treated like a monitoring well without a pump. One limitation is that in most cases the in-place pump is “hard” mounted, that is, the pump is suspended in the well at a pre-selected depth and cannot be moved up or down during purging and sampling. In these cases, well volumes are calculated, parameters are measured and the well is sampled from the pump discharge, after volume removal and parameter conditions have been met.

In the case of the other types of wells, i.e., municipal, industrial and residential supply wells, however, not enough is generally known about the construction aspects of the wells to apply the same criteria as used for monitoring wells, i.e., 3 to 5 well volumes. The volume to be purged in these situations, therefore, depends on several factors: whether the pumps are running continuously or intermittently and whether or not any storage/pressure tanks are located between the sampling point and the pump. The following considerations and procedures should be followed when purging wells with in-place plumbing under the conditions described.

3.3.2.1 Continuously Running Pumps

If the pump runs more or less continuously, no purge (other than opening a valve and allowing it to flush for a few minutes) is necessary. If a storage tank is present, a spigot, valve or other sampling point should be located between the

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pump and the storage tank. If not, locate the valve closest to the tank. Measurements of pH, specific conductance, temperature, and turbidity are recorded at the time of sampling.

3.3.2.2 Intermittently or Infrequently Running Pumps

If the pump runs intermittently or infrequently, best judgment should be utilized to remove enough water from the plumbing to flush standing water from the piping and any storage tanks that might be present. Generally, under these conditions, 15 to 30 minutes will be adequate. Measurements of pH, specific conductance, temperature and turbidity should be made and recorded at intervals during the purge and the final measurements made at the time of sampling should be considered the measurements of record for the event.

3.3.3 Temporary Monitoring Wells

3.3.3.1 General Considerations

Procedures used to purge temporary ground water monitoring wells differ from permanent wells because temporary wells are installed for immediate sample acquisition. Wells of this type may include standard well screen and riser placed in boreholes created by hand augering, power augering, or by drilling. They may also consist of a rigid rod and screen that is pushed, driven, or hammered into place to the desired sampling interval, such as a direct push Wellpoint®, a Geoprobe® Screen Point 15/16 sampler or a Hydropunch® sampler. As such, the efforts to remove several volumes of water to replace stagnant water do not necessarily apply because stagnant water is not present. It is important to note, however, that the longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply, to the extent possible, standard permanent monitoring well purging criteria to it to re-achieve aquifer conditions.

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, resulting primarily in increased turbidity. Therefore, the goal of purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure. Low turbidity conditions in these types of wells that are completed within the limit of suction are typically and routinely achieved by the use of low-flow/low stress purging techniques using variable speed peristaltic pumps.

3.3.3.2 Purging When Water Level Is Within Limit of Suction

In situations where the elevation of the top of the water column is within the limit of suction (no greater than about 25 feet head difference between the pump and the water level), a variable speed peristaltic pump may be used to purge

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temporary wells. Enough tubing is deployed to reach the bottom of the temporary well screen. At the onset of purging, the tubing is slowly lowered to the bottom of the screen and is used to remove any formation material which may have entered the well screen during installation. This is critical to ensuring rapid achievement of low turbidity conditions. After the formation material is removed from the bottom of the screen, the tubing is slowly raised through the water column to near the top of the column. The tubing can be held at this level to determine if the pump rate is drawing down the water level in the well. If the water level remains the same, secure the tubing at the surface to maintain this pumping level.

If drawdown is observed on initiation of pumping, reduce the pump speed and attempt to match the drawdown of the well. Sustained pumping at these slow rates will usually result in a relatively clear, low turbidity sample. If the drawdown stabilizes, maintain that level, however, if it continues to lower, "chase" the water column until the well is evacuated. In this case, the recovered water column may be relatively free of turbidity and can be sampled. It may take several episodes of recovery to provide enough volume for a complete sample.

3.3.3.3 Purging When Water Level Is Greater Than Limit of Suction

In situations where the elevation of the water table is greater than the limit of suction, peristaltic pumps cannot be used to purge temporary wells. If the temporary well is a ScreenPoint15® sampler with small diameter probe rod riser, the only practical choices for water removal are a small diameter bailer, a small diameter bladder pump or an inertial pump. If the well is to be used strictly for VOC screening, it may be acceptable to use the bailer to bail as much sediment from the well as possible prior to sampling. If metals are the analytes of concern, the bladder pump is the best choice for lowering the turbidity of the water column prior to sampling, followed next by the inertial pump. For larger diameter temporary wells, two-inch diameter or greater, bailers and the Grundfos® RediFlo2 may be used although excessive silt or other "fines" may present problems with the operation of the pump.

3.3.3.4 Considerations for Direct Push Groundwater Sampling

With many of the direct push sampling techniques, purging is either not practical or possible, therefore, no purging is conducted. The sampling device is simply pushed or driven to the desired depth and opened and the sample is collected and retrieved. As a result, some samples collected in this way may not be satisfactory or acceptable for certain analyses, i.e., the subject procedure may yield a turbid sample that is not appropriate for metals analyses.

3.4 Field Care of Purging Equipment

New plastic sheeting should be placed on the ground surface around the well casing to prevent contamination of the pumps, hoses, ropes, etc., in the event they accidentally

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come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event. It is preferable that hoses used in purging that come into contact with the ground water be kept on a spool or contained in a large wash tub lined with plastic sheeting, both during transportation and during field use, to further minimize contamination by the transporting vehicle or the ground surface.

Careful consideration shall be given to using submersible pumps to purge wells which are excessively contaminated with oily compounds, because it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When wells of this type are encountered, alternative purging methods, such as bailers, should be considered.

3.5 Investigation Derived Waste

Purging generates quantities of purge water or investigation derived waste (IDW), the disposition of which must be considered. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for guidance on management or disposal of this waste.

4 Groundwater Sampling Methods – Sampling

4.1 General

Sampling is the process of obtaining, containerizing, and preserving (if required) a ground water sample after the purging process is complete. Non-dedicated pumps for sample collection generally should not be used. Many pumps are made of materials such as brass, plastic, rubber, or other elastomeric products which may cause chemical interferences with the sample. Their principle of operation may also render them unacceptable as a sample collection device. It is recognized that there are situations, such as industrial or municipal supply wells or private residential wells, where a well may be equipped with a dedicated pump from which a sample would not normally be collected. Discretion should always be used in obtaining a sample.

4.2 Sampling Wells With In-Place Plumbing

Samples should be collected following purging from a valve or cold water tap as near to the well as possible, preferably prior to any storage/pressure tanks or physical/chemical treatment system that might be present. Remove any hose that may be present before sample collection and reduce the flow to a low level to minimize sample disturbance, particularly with respect to volatile organic constituents. Samples should be collected directly into the appropriate containers as specified in the ASBLOQAM. It may be necessary to use a secondary container, such as a clean 8 oz. or similar size sample jar or a stainless steel scoop, to obtain and transfer samples from spigots with low ground clearance. Also, refer to the discussion in the SESD Operating Procedure for Potable Water Supply Sampling (SESDPROC-305), Sec. 4.2, Potable Water Samples Collected from Wells with In-Place Plumbing. Potable well measurements for pH, specific conductance and turbidity and possibly temperature, if warranted, should be recorded at the time of sample collection.

4.3 Sampling Wells Without Plumbing, Within the Limit of Suction

4.3.1 Equipment Available

The pump of choice for sampling ground water within the limit of suction is the variable-speed peristaltic pump. Its use is described in the following sections. Other acceptable alternatives that may be used under these conditions are the RediFlo2® electric submersible pump (with Teflon® tubing) and a closed-top Teflon® bailer.

4.3.1.1 Peristaltic Pump, Direct from Pump Head Tubing

Samples for some constituents, primarily inorganic analytes such as metals and cyanide, may be collected directly from the pump head tubing. This method is acceptable under the following conditions:

- The pump head tubing must be changed between sampling locations;

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- The pump head tubing must be either be certified clean according to SESD's internal quality control program described in Section 3.2 of the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) or
- An equipment rinsate blank is collected by pumping de-ionized water through a piece of the tubing.

4.3.1.2 *Peristaltic Pump/Vacuum jug*

It is not acceptable to collect samples for organic compound analyses through the flexible tubing used in the pump head. When collecting samples for organic compound analyses it is necessary to use a vacuum container, placed between the pump and the well for sample collection. The following step-by-step procedures describe the process of sampling with a peristaltic pump and vacuum jug (see note following these procedures for collection of VOC samples):

1. Disconnect the purge tubing from the pump. Make sure the tubing is securely attached to the protective casing or other secure object.
2. Insert the tubing into one of the ferrule nut fittings of a Teflon® vacuum container transfer cap assembly.
3. Place a suitable length of Teflon® tubing between the remaining transfer cap assembly ferrule nut fitting and the vacuum side of the flexible tubing in the peristaltic pump head. Securely hand-tighten both fittings.
4. Turn the pump on. Water should begin to collect in the transfer container (typically a 1-liter sample container) within a few minutes. If water does not begin to flow into the container within several minutes, check the transfer cap fittings and make sure the assembly is tightly attached to the container. It may be necessary to tighten the ferrule nuts with a wrench or pliers to achieve a vacuum in the system, particularly when approaching the maximum head difference between the pump and water table (limit of suction).
5. When the transfer container is nearly full, turn off the pump, remove the transfer cap assembly, and pour the sample into the appropriate containers. Because the 1-liter containers used by the Branch are rinsed with nitric acid during cleaning, they cannot be used for collecting samples to be analyzed for nitrogen sensitive parameters.
6. If additional sample volume is needed, replace the transfer cap assembly, turn the pump on, and collect additional volume. The use of Teflon® valves or ball check devices to retain the water column in the sample delivery tubing during the transfer phase, when large volumes of sample are required, is acceptable. These devices, however, must be constructed so that they may be completely disassembled and cleaned according to the procedures in SESD

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Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

7. When sampling is completed, all Teflon® tubing should be discarded.

NOTE: Samples for volatile organic compound analyses cannot be collected using this method. If samples for VOC analyses are required, they must be collected with a Teflon® or stainless steel bailer or by other approved methods, such as the “soda straw” method. The “soda straw” method involves allowing the tubing to fill, by either lowering it into the water column (A) or by filling it via suction applied by the pump head (B). If method (A) is used, the tubing is removed from the well after filling and the captured sample is allowed to drain into the sample vial. If method (B) is used, after running the pump and filling the tubing with sample, the pump speed is reduced and the direction reversed to push the sample out of the tubing into the vials. Avoid completely emptying the tubing when filling the sample vials when using method (B) to prevent introducing water that was in contact with the flexible pump head tubing. Either method is repeated, as necessary, until all vials are filled.

4.3.1.3 RediFlo2® Electric Submersible Pump (with Teflon® Tubing)

After purging has been accomplished with RediFlo2® electric submersible pump, the sample may be obtained directly from the pump discharge, provided that Teflon® tubing was used for the sample delivery line. The discharge rate of the pump should be reduced during volatile organic compound sample collection to minimize sample disturbance. Note, if the RediFlo2® electric submersible pump is used for sampling, the pump must undergo a full external and internal cleaning. In addition, pump rinsate blanks must be collected, at the appropriate frequency, to demonstrate that the pump has been adequately cleaned between wells.

4.3.1.4 Bailers

New bailer rope should be attached to the bailer via a Teflon® coated stainless steel wire. (If a bailer was used to purge the well, it may also be used to sample the well and new bailer rope is not required between purging and sampling). The bailer should be gently immersed in the top of the water column until just filled. At this point, the bailer should be slowly removed and the contents emptied into the appropriate sample containers.

4.4 Sampling Wells without Plumbing, Exceeding the Limit of Suction

All methods described previously in Section 4.3.2.1.3, RediFlo2® Electric Submersible Pumps, and Section 4.3.2.1.4, Bailers, are suitable sample methods where the water table is too deep to consider the use of a peristaltic pump for sampling.

4.5 Micro-Purge or No Purge Sampling Procedures

The Micro-Purge or No Purge sampling procedures are usually employed when it necessary to keep purge volumes to an absolute minimum. Among the Micro-Purge or No Purge procedures that might be employed are:

- Low pump rate sampling with peristaltic or submersible pumps (typical Micro-Purge sampling),
- HydraSleeve™ or
- Passive diffusion bag (PDB) sampling

The use of these procedures is acceptable only when the site hydrogeology is well understood, with respect to the hydraulic conductivity of geologic materials within the well screen interval. The underlying assumption, when employing these procedures, is that the formation in which the well is screened has a high hydraulic conductivity ($K > 10^{-5}$ cm/sec, for example), resulting in a state of equilibrium existing between the water standing in the screened interval and the formation water in which the well is screened. In this situation, the well is considered to be in a perpetually “purged” state and purging is not required.

These procedures are generally impractical for SESD to implement because of the general lack of hydrogeologic information for the sampled wells and the real necessity, in some cases, that the pumps be pre-deployed to overcome issues related to turbidity resulting from pump placement prior to sampling.

4.5.1 Sampling with Pumps

The peristaltic pump tubing or intake point of the submersible pump is placed in the approximate mid-portion of the screened interval of the well or other interval selected by the field team leader. If turbidity and its impact on metals analyses are a concern, a period of time sufficient should be allowed to mitigate effects of pump or tubing placement. After it has been determined that sampling may proceed, the pump is turned on and operated at a rate that does not cause significant drawdown of the water column, as measured using a water level sounder. During sampling, sufficient water to supply enough volume for the analytes of concern and the purge parameters is pumped. Purging should continue until purge parameters stabilize, generally three consecutive stable sets of readings, before samples are collected.

4.5.2 HydraSleeves™

HydraSleeves™ are grab sampling devices that are deployed in a closed configuration then opened in the desired interval for sample collection. The following is a summary of its operation:

1. Sampler placement - Reusable weight is attached and the HydraSleeve™ is lowered and placed at the desired position in the well screen. In-situ water pressure keeps the reed valve closed, preventing water from entering the sampler. Well is allowed to return to equilibrium.

2. Sample collection - The reed valve opens to allow filling when the sampler is moved upward faster than 1 foot per second, either in one continuous upward pull or by cycling the sampler up and down to sample a shorter interval. There is no change in water level, and only minimal agitation during collection.
3. Sample retrieval - When the flexible sleeve is full, the reed valve closes and the sampler can be recovered without entry of extraneous overlying fluids. Samples are removed by puncturing the sleeve with the pointed discharge tube and draining the contents into containers for sampling or field measurement.

4.5.3 Passive Diffusion Bags

Passive diffusion bag (PDB) samplers are bags comprised of low-density polyethylene (LDPE) plastic and containing analyte-free water, preferably with no headspace. The bags are deployed, with stainless steel weights, to the desired sample interval and are allowed to equilibrate with the water at the point of deployment in the well. A deployment period of a minimum of 14 days is recommended to ensure equilibration prior to removal.

After 14 days, the bags are opened with a puncture device or other cutting implement and the contents transferred to containers for sampling or field measurement.

4.5.4 General Considerations for Micro-Purge or No-Purge Sampling

When using the Micro-Purge method, it may be advisable to deploy the tubing or pump in advance of sample collection. Introducing the tubing or pump into the screened interval is likely to dislodge sediment and other fines that have settled or bridged on the well screen material and the gravel pack media behind the screen. If sampling is conducted immediately, turbidity issues may render this method impractical from a parameter stability standpoint.

HydraSleeves™ and PDBs must be evaluated for appropriateness for analytes of concern.

4.6 Sample Preservation

After sample collection, all samples requiring preservation must be preserved as soon as practical. Consult the ASBLOQAM for the correct preservative for the particular analytes of interest. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

4.7 Special Sample Collection Procedures

4.7.1 Trace Organic Compounds and Metals

Special sample handling procedures should be instituted when trace contaminant samples are being collected. All sampling equipment, including pumps, bailers, water level measurement equipment, etc., which comes into contact with the water in the well must be cleaned in accordance with the cleaning procedures described in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206). Pumps should not be used for sampling unless the interior and exterior portions of the pump and the discharge hoses are thoroughly cleaned. Blank samples should be collected to determine the adequacy of cleaning prior to collection of any sample using a pump other than a peristaltic pump.

4.7.2 Order of Sampling with Respect to Analytes

In many situations when sampling permanent or temporary monitoring wells, an adequate purge, with respect to turbidity, is often difficult to achieve. Removal and insertion of equipment after the purge and prior to actual sampling may negate the low turbidities achieved during purging and elevate turbidity back to unacceptable levels. For this reason, it is important that special efforts be used to minimize any disturbance of the water column after purging and to collect the aliquot for metals first. Therefore, the preferred order of sampling is metals first, followed by other inorganic analytes, extractable organic compounds and volatile organic compounds.

4.7.3 Filtering

As a standard practice, ground water samples will not be filtered for routine analysis. Filtering will usually only be performed to determine the fraction of major ions and trace metals passing the filter and used for flow system analysis and for the purpose of geochemical speciation modeling. Filtration is not allowed to correct for improperly designed or constructed monitoring wells, inappropriate sampling methods, or poor sampling technique.

When samples are collected for routine analyses and are filtered, both filtered and non-filtered samples will be submitted for analyses. Samples for organic compounds analysis should not be filtered. Prior to filtration of the ground water sample for any reason other than geochemical speciation modeling, the following criteria must be demonstrated to justify the use of filtered samples for inorganic analysis:

1. The monitoring wells, whether temporary or permanent, have been constructed and developed in accordance with the SESD Guidance Document, Design and Installation of Monitoring Wells (SESDGUID-001).

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2. The ground water samples were collected using sampling techniques in accordance with this section, and the ground water samples were analyzed in accordance with USEPA approved methods.
3. Efforts have been undertaken to minimize any persistent sample turbidity problems. These efforts may consist of the following:
 - Redevelopment or re-installation of permanent ground water monitoring wells.
 - Implementation of low flow/low stress purging and sampling techniques.
4. Turbidity measurements should be taken during purging and sampling to demonstrate stabilization or lack thereof. These measurements should be documented in the field notes. If the ground water sample appears to have either a chemically-induced elevated turbidity, such as would occur with precipitate formation, or a naturally elevated colloid or fine, particulate-related turbidity, filtration will not be allowed.

If filtration is necessary for purposes of geochemical modeling or other **pre-approved** cases, the following procedures are suggested:

1. Accomplish in-line filtration through the use of disposable, high capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus. The high capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration. If a membrane filter is utilized, a minimum diameter of 142 mm is suggested.
2. Use a 5 µm pore-size filter for the purpose of determining the colloidal constituent concentrations. A 0.1 µm pore-size filter should be used to remove most non-dissolved particles.
3. Rinse the cartridge or barrel-type filter with 500 milliliters of the solute (groundwater to be sampled) prior to collection of sample. If a membrane filter is used, rinse with 100 milliliters of solute prior to sample collection.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloiddally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from the following: 1) Metals in Ground Water: Sampling Artifacts and Reproducibility; 2) Filtration of Ground Water Samples for Metals Analysis; and 3) Ground Water Sampling - A Workshop Summary. See Section 1.4, References, for complete citation for these documents.

Bacterial Sampling

Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in the following: 1) Sampling for Organic Chemicals and Microorganisms in the Subsurface; 2) Handbook for Evaluating Water Bacteriological Laboratories; and 3) Microbiological Methods for Monitoring the Environment, Water and Wastes. See Section 1.4, References, for complete citation for these documents.

4.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect ground water samples shall be cleaned as outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

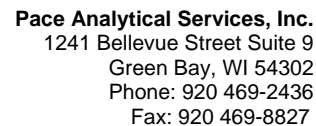
4.9 Auxiliary Data Collection

During ground water sample collection, it is important to record a variety of ground water related data. Included in the category of auxiliary data are water levels measured according to the SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105), well volume determinations (Section 3.1.1, Purging and Purge Adequacy), pumping rates during purging (see below), and occasionally, drillers or boring logs. This information should be documented in the field records.

4.9.1 Well Pumping Rate – Bucket/Stop Watch Method


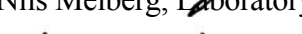

The pumping rate for a pump can be determined by collecting the discharge from the pump in a bucket of known volume and timing how long it takes to fill the bucket. The pumping rate should be in gallons per minute. This method shall be used primarily with pumps with a constant pump rate, such as gasoline-powered or electric submersible pumps. Care should be taken when using this method with some battery-powered pumps. As the batteries' charge decreases, the pump rate also decreases so that pumping rate calculations using initial, high pump rates may be erroneously high. If this method is used with battery-powered pumps, the rate should be re-checked frequently to ensure accuracy of the pumping rate calculations.

ATTACHMENT B1



REFERENCE METHODS: EPA Method 3510C

LOCAL SOP NUMBER:	S-GB-O-053-Rev.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-053-Rev.02
SOP TEMPLATE NUMBER:	SOT-ALL-O-003-Rev.01

	06/27/14
Nils Melberg, Laboratory General Manager	Date
	6/26/14
Kate Grams, Laboratory Quality Manager	Date
	06/20/14
Chris Haase, Department Manager	Date

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

- 1.1. The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from aqueous samples in a separatory funnel while meeting the requirements specified in EPA method 3510C.

2. Summary of Method

- 2.1. A measured volume of sample (100 mL and 1 liter volumes) are serially extracted with solvent in a separatory funnel. Some extractions also require the monitoring and adjusting of the pH of the sample. The extract is separated from the sample and is concentrated, followed by cleanup or analysis.

3. Scope and Application

- 3.1. **Personnel:** This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of separatory funnel equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.
- 3.2. **Parameters:** This procedure is applicable for the extraction of semi-volatile compounds (BNAs), polynuclear aromatic hydrocarbons (PAHs), pesticides, PCBs and total petroleum hydrocarbons (TPH). The compounds can be found in the appropriate analytical SOPs listed below.
- 3.2.1. The most current revision of analytical SOPs using this extraction procedure includes:

S-GB-O-049	Determination of Semivolatile Organics by GC/MS
S-GB-O-050	Determination of Semivolatile Organics by GC/MS (Selective Ion Monitoring)
S-GB-O-026	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography
S-GB-O-027	Analysis of Organochlorine Pesticides by Gas Chromatography
S-GB-O-023	Total Petroleum Hydrocarbons

4. Applicable Matrices

- 4.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from aqueous samples.

5. Limits of Detection and Quantitation

- 5.1. Not applicable to this SOP.

6. Interferences

- 6.1. Solvents, reagents and glassware can all contribute to compound artifacts or raised baselines; both conditions that can affect chromatography. Analyzing method blanks is

therefore crucial in determining the presence of contaminants.

- 6.2.** Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.
- 6.3.** Extracts that exhibit interferences can be run through a cleanup procedure (see EPA method 3600). Before using a cleanup method, the analyst should run a series of calibration standards through the procedure to ensure that the elution order of compound remains the same and that no new interference has been introduced by the cleanup method. The most current revision of cleanup SOPs that can be used for this extraction procedure include:

S-GB-O-032	Gel Permeation Chromatography
S-GB-O-034	Sulfuric Acid Cleanup
S-GB-O-036	Florisil Cleanup for PCBs
S-GB-O-037	Florisil Cartridge Cleanup
S-GB-O-038	Silica Gel Cleanup for Organic Analysis
S-GB-O-039	Copper Cleanup for the Removal of Sulfur from PCB Samples

7. Sample Collection, Preservation, Shipment and Storage

- 7.1.** Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

Table 7.1 – Sample Collection, Preservation, Storage, and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid (preferably 1L wide-mouth).	None	≤6°C	BNA, PAH, Pesticide and TPH-Diesel samples must be extracted within 7 days of collection and analyzed within 40 days of extraction. In lieu of project specific requirements, PCB samples must be extracted within 365 days of collection and must be analyzed within 365 days of extraction.
Aqueous (Low Volume Extraction-PAH)	100mL vials with Teflon lined lid.	None	≤6°C	Samples must be extracted within 7 days of collection and must be analyzed within 40 days of extraction.
TCLP and SPLP	One (1) 1-Liter Amber Glass	None	≤6°C	TCLP and SPLP Leachates must be tumbled within 14 days of collection. The leachate solvent must be extracted within 7 days of the start of the tumbling process.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Instrumentation

Equipment	Vendor	Model /
Lab-Line Automated Separatory Funnel Extractor	Barnstead	Model #1600
Concentrator Water Bath	Fisher	S-EVAP
Turbo Vaps II	Zymark	Zw 8002

9.2. General Supplies

Item	Vendor	Model / ID	Catalog #	Description
Self-venting Separatory Funnels	Fisher		NC9802352	Glass, able to hold 2L; with PTFE stopcocks and Teflon lids
Analytical Balance	Ohaus	Model # AR 5120		Capable of reading to 0.01g
Glass beakers	Fisher			
Glass Autosampler Vials	MG Scientific	V300-3 / V300-20N		2.0 mL with Teflon-line
Micro-syringes	Hamilton			10-μL, 25-μL, 50-μL, 100-μL, 250-μL, 500-μL, and 1,000-μL, as needed, Hamilton or equivalent.
Glass funnels	Fisher			
Whatman #41 Filters	Fisher	1441-185		185 mm
Glass Stirring Rods	Fisher			
Kuderna-Danish flasks	HGF Scientific	192006-03		500mL with ground glass joints
Kuderna Danish concentrator tubes	HGF Scientific	192010-12		10mL, graduated, with ground glass fitting
Snyder columns	HGF Scientific	192002-DP1/ 192002-M2B		2-ball and 3-ball varieties
Keck clips to hold KD glassware together	Fisher	NO19	05-880D	Keck Clip #19
Boiling Chips	Fisher	09-191-20		450 g
pH test strips	CTL		921-10	Wide Range 0-14

Item	Vendor	Model / ID	Catalog #	Description
Dispensing Pipettes	Fisher			Adjustable over range of 50.0 to 100.0 mL, one for each solvent
Wash bottles, PTFE	Fisher			One for each solvent
Powder funnels	Fisher			
Glass wool 8 micron	Fisher		551940	
Disposable Pasteur pipettes	MG	P200-1 5 3/4" P200-2 9"		
125 mL FEP Separatory funnels	Fisher		4301-0125	FEP, able to hold 125 mL with PTFE stopcocks
25 mL condenser	HGF		192010	25 mL
Snyder column	HGF		192040-02-24	24/40 joints

10.

10. Reagents and Standards

10.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular, baked at 400°C for 4 hours before use.	MG Scientific / catalog # 8024-24
Methylene Chloride	Extraction solvent	MG Scientific / catalog # 9266-8P
Acetone	Extraction solvent	MG Scientific / catalog # 010-4
Hexane	Exchange solvent (pesticides and PCBs)	MG Scientific / catalog # 9262-8P
Sulfuric Acid, Conc.	Concentrated	Fisher / catalog # 9681-33
Sulfuric Acid Solution 1:1	Add 400 mL conc. Sulfuric Acid to 400 mL Reagent Water	
Sodium Hydroxide pellets		Fisher / catalog # 5318-3
Sodium Hydroxide Solution (10N)	Dissolve 400g sodium hydroxide pellets into 1L of reagent water	

10.2 Analytical Standards

10.2.1 Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2: Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains all target analytes.	Same solution can be used for the LCS and MS/MSD

10.2.2 Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Surrogate Standards

Standard	Concentration	Manufacturer	Catalog #	Storage
B/N Surrogate Mix for PAHs	1000 ug/mL	Restek	31002	Refrigerator $\leq 6^{\circ}\text{C}$
B/N Sur Mix for Low Volume PAH	200 ug/mL	Restek	31002	Refrigerator $\leq 6^{\circ}\text{C}$
B/N Sur Mix for BNAs	5000 ug/mL	Restek	31082	Refrigerator $\leq 6^{\circ}\text{C}$
Acid Surrogate Mix for BNAs	7500 ug/mL	Restek	31083	Refrigerator $\leq 6^{\circ}\text{C}$
Equity Pesticide Surrogate Spike Mix	200 $\mu\text{g/mL}$ each TMX and DCB in Acetone	Supelco or Restek	5-05935 or 32457	Refrigerator $\leq 6^{\circ}\text{C}$
O-Terphenyl Surrogate	10000 ug/mL	Restek	31097	Refrigerator $\leq 6^{\circ}\text{C}$

LCS and MS Standards

Standard	Concentration	Manufacturer	Catalog #	Storage
PAH Standard	500 ug/mL	Accustandard	M-160-FL-R-5X	Refrigerator $\leq 6^{\circ}\text{C}$
PAH Low Volume Std	200 ug/mL	Accustandard	M-160-FL-R-5X	Refrigerator $\leq 6^{\circ}\text{C}$
BNA Std - 70 Component Custom LCS Mix	200 ug/mL	Supelco	861389-U	Refrigerator $\leq 6^{\circ}\text{C}$
BNA Standard - n-nitrosodiphenylamine	5000 ug/mL	Supelco	46702-U	Refrigerator $\leq 6^{\circ}\text{C}$
Pesticide Standard Mix A	5-50 $\mu\text{g/mL}$ in Hexane:Toluene (98:2)	Supelco or equivalent	4-8796	Refrigerator $\leq 6^{\circ}\text{C}$
Pesticide Standard Mix B	5 – 50 $\mu\text{g/mL}$ in Hexane:Toluene (99:1)	Supelco or equivalent	4-8196	Refrigerator $\leq 6^{\circ}\text{C}$
Aroclor 1260	1000 $\mu\text{g/mL}$ in isooctane	Supelco or equivalent	4-4809	Room Temperature
Aroclor 1016*	1000 $\mu\text{g/mL}$ in	Supelco or	4-8097	Room Temperature

Standard	Concentration	Manufacturer	Catalog #	Storage
	isooctane	equivalent		
Aroclor 1242	1000ug/mL in isooctane	Supelco or equivalent	48053-u	Room Temperature
Aroclor 1254	1000ug/mL in isooctane	Supelco or equivalent	4-4808	Room Temperature
Toxaphene Standard	1000µg/mL in Hexane	Restek or equivalent	32005	Refrigerator ≤6°C
Diesel Fuel #2	50000µg/mL	Restek or equivalent	32158	Room Temperature

*South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS and MSD

10.2.3 Preparation Procedures PAHs by SW-846 8270C-SIM

- Working Spiking Standard Preparation**

Table 10.4 Preparation of PAH LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
PAH Working Spike Solution	PAH Standard	200µL	500mL of methylene chloride	0.2µg/mL	1000µL
LV PAH Working Spike Solution	PAH Standard	200µL	500mL of methylene chloride	0.2µg/mL	100µL

- Working Surrogate Standard Preparation**

Table 10.5: Preparation of PAH Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Working PAH Surrogate Stock Solution	Restek B/N Mix, Cat. # 31082	100µL	500mL of methylene chloride	0.2µg/mL	1000 µL
LV PAH Surrogate Stock Solution	Restek B/N Mix, Cat #31082	100µL	500mL of methylene chloride	0.2µg/mL	100 µL

BNAs by SW-846 8270C

- Working Spiking Standard Preparation**

Table 10.6: Preparation of BNA LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
BNA Working Spike Solution	BNA Standard - 70 Component Custom LCS Mix	250µL	250µL	50µg/mL	250µL
	BNA Standard - n-nitrosodiphenylamine	10µL	10µL	50µg/mL	10µL

- Working Surrogate Standard Preparation**

Table 10.7 Preparation of BNA Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
BNA Surrogate Spike Solution	Restek Acid Surrogate Mix for BNAs	5000µL	500mL of Methylene chloride	75µg/mL Acids	1.0 mL
	Restek B/N Surrogate Mix for BNAs	5000µL		50µg/mL Base/Neutrals	

Pesticide by SW-846 8081

- Working Spiking Standard Preparation**

Table 10.8: Preparation of Pesticide LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide Matrix Spike Solution	Pesticide Standard Mix A and Mix B	4000µL of each	50mL of Acetone	0.4 – 4.0µg/mL	1000µL
Toxaphene Matrix Spike Solution	Toxaphene Standard	5000µL	100mL of Acetone	50µg/mL	800µL

- **Working Surrogate Standard Preparation**

Table 10.9: Preparation of Pesticide Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide/PCB Surrogate Solution	Equity Pesticide Surrogate Spike Mix	5000µL	500mL of Acetone	2.0µg/mL	500µL

PCB by SW-846 8082

- **Working Spiking Standard Preparation**

Table 10.10: Preparation of PCB LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
PCB Matrix Spike Solution	Aroclor 1260	1000µL	200mL of Acetone	5.0µg/mL	1000µL
PCB Matrix Spike Solution	Aroclor 1016*	1000µL	200mL of Acetone	5.0µg/mL	1000µL
PCB Matrix Spike Solution	Aroclor 1242	4000µL	200mL of Acetone	20.0µg/mL	250µL
PCB Matrix Spike Solution	Aroclor 1254	1000µL	200mL of Acetone	5.0µg/mL	1000µL

*South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS and MSD samples.

- **Working Surrogate Standard Preparation**

Table 10.11: Preparation of Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide/PCB Surrogate Solution	Equity Pesticide Surrogate Spike Mix	5000µL	500mL of Acetone	2.0µg/mL	500µL

TPH by SW-846 8015B

- **Working Spiking Standard Preparation**

Table 10.12: Preparation of TPH LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Working Diesel Spike Solution	Restek Diesel Fuel #2 or equivalent	2mL	100mL of Methylene Chloride	1000µg/mL	500µL

- **Working Surrogate Standard Preparation**

Table 10.13: Preparation of Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
TPH Surrogate Solution	O-Terphenyl Surrogate	2500µL	250mL of Methylene Chloride	100µg/mL	500µL

11. Calibration

11.1 Not applicable to this SOP.

12. Procedures

- 12.1** See the latest revision of Pace SOP S-GB-O-015, *Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics* (most current revision or replacement) for the specifics on glassware cleaning.
- 12.2** Inspect all required glassware to ensure it is clean and dry. Set up each extraction mixer with as many as four, 2L shaker funnels, including caps and stopcocks; assemble 500mL KD apparatus; prepare water funnels with glass wool and sodium sulfate; and pre-rinse all glassware with methylene chloride.
- 12.3** To perform low volume PAH extraction set up as many as 12 - 125mL FEP separatory funnels including caps and stopcocks. Rinse a 25 mL condenser with Methylene Chloride.
- 12.4** Check the pH of any sample aliquots that will be analyzed for Methods 8270, 8081, 8082, or 8015 by removing a few drops with a disposable pipette for application to pH strips. Record result.
- 12.5** Mark the sample level on the container, pour the entire contents into separatory funnel and adjust pH accordingly. Add the appropriate working spike and working

surrogate solutions as outlined in 12.6. Rinse the container with 60 mL of methylene chloride and pour into separatory funnel. For low volume extraction method rinse the container with 6 mLs of methylene chloride and pour into separatory funnel. Narrow-mouth bottles contain 1060 mL of sample. Wide-mouth bottles contain 1000 mL of sample. Containers that were not received full have the sample level meniscus marked as stated above, and after pouring of the sample into the separatory funnel and rinsing with methylene chloride, the container is filled to the mark with water. This water is then poured into a 1000 mL graduated cylinder and the sample volume recorded in the extraction log. If the sample container contains sediment on the bottom of the container, care is taken not to disturb and pour this material into the separatory funnel.

- 12.5.1** If high concentrations are anticipated, a smaller sample aliquot may be diluted to 1000 mL with nano pure water prior to extraction.
- 12.5.2** For TCLP and SPLP Leachates 200 mL of sample is measured with a Class A Graduated Cylinder and the contents poured into a separatory funnel. An additional 800 mL of Nanopure or Millipore water is also added to the separatory funnel. The volumes are recorded in the SW846 3510C Extraction Log. Add working spike and working surrogate solutions as outlined in 12.6. Rinse the graduated cylinder with 60 mL of methylene chloride and pour into the separatory funnel.
- 12.6** For each batch of 20 samples, or less, prepare two additional sample aliquots. One will serve as a MS and the second as a MSD. As an alternative if no sample in the batch has sufficient volume available for both a MS and MSD, prepare a second aliquot of reagent water for a Laboratory Control Spike Duplicate. Also prepare 2 aliquots of reagent water to serve as the Method Blank and LCS.
 - 12.6.1** For TCLP and SPLP samples a MS must be prepared for each sample matrix type. (For example sand is determined to be a different matrix type than sludge, and a separate matrix spike must be prepared for each one.) This information is recorded on the TCLP/SPLP extraction paperwork, and is provided the organic preparation personnel prior to extraction. The organic preparation personnel also record this information on the SW846 3510C Extraction Log.
- 12.7** Add Working Spike and Working Surrogate Solutions as follows:
 - 12.7.1** PAH Analysis – Add 1.0 mL of 0.2 ug/mL Working Spike Solution (10.4) to the LCS, LCSD, MS, and MSD. Add 1.0 mL of the 0.2 ug/mL Working Surrogate Solution (10.5) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
 - 12.7.2** PAH Low Volume Extraction Analysis- Add 100 µL of 0.2 ug/mL Working Spike Solution (10.4) to the LCS, LCSD, MS, and MSD. Add 100 µL of the 0.2 ug/mL Working Surrogate Solution (10.5) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
 - 12.7.3** BNA Analysis – Add 250 uL of Supelco 70 Component Custom LCS Spike Mix, 200 ug/mL (10.6) and 10 uL of Supelco n-Nitrosodiphenylamine, 5000 ug/mL

- (10.6) to the LCS, LCSD, MS, and MSD. Add 1.0 mL of the Working Surrogate Solution, 75 ug/mL Acids, 50 ug/mL Base/Neutral (10.7) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
- 12.7.4** Pesticide Analysis – Add 1.0 mL of 0.4-4.0 ug/mL Working Spike Solution (10.8) to the LCS, LCSD, MS, and MSD. Add 500 uL of the 2.0 ug/mL Working Surrogate Solution (10.9) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
- 12.7.5** PCB Analysis – Add 1.0 mL of 5.0 ug/mL or 250µL of a 20.0ug/mL Working Spike Solution (10.10) to the LCS, LCSD, MS, and MSD. Add 500 uL of the 2.0 ug/mL Working Surrogate Solution (10.11) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
- 12.7.6** Toxaphene Analysis – Add 800 uL of 50 ug/mL Working Spike Solution (10.8) to the LCS, LCSD, MS, and MSD. Add 500 uL of the 2.0 ug/mL Working Surrogate Solution (10.9) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
- 12.7.7** TPH analysis – Add 500 µL of 1000 µg/mL Working Spike Solution (10.12) to the LCS, LCSD, MS, and MSD. Add 500 µL of 100 µg/mL Working Surrogate Solution (10.13) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.
- 12.8** Adjust sample aliquots that will be analyzed for Method 8270 to pH <2 with 1:1 sulfuric acid solution. Adjust sample aliquots that will be analyzed for Methods 8081A or 8082 to pH 5-9 with 1:1 (v/v) sulfuric acid or 10N sodium hydroxide solution.
- 12.9** Add 60 mL of dichloromethane to each sample aliquot. If the entire content of a sample bottle is extracted, rinse the container with the dichloromethane before adding to the separatory funnel. Then pour tap water into the container to the mark, and measure the tap water volume with the graduated cylinder.
- 12.10** Low volume extraction for PAH SIM will add 6 mL of dichloromethane to each sample aliquot. If the entire content of a sample bottle is extracted, rinse the container with the dichloromethane before adding to the separatory funnel. Then pour tap water into the container to the mark, and measure the tap water volume with the graduated cylinder.
- 12.11** Seal the separatory funnel and set the Lab-Line extraction mixer to shake for 3.0 minutes at a speed of 30 cycles per minutes. After shaking allow the sample and extract to settle for at least 10 minutes.
- 12.12** If an emulsion is observed at the water-solvent interface with a volume more than 1/3 of the solvent layer, mechanical techniques should be used to complete separation. Mechanical techniques include stirring, filtration through a plug of glass wool, or centrifuging. If 80% of the solvent cannot be recovered, an alternative extraction method may be required.
- 12.13** Drain and dry the extract through powder funnels containing glass wool and

anhydrous sodium sulfate. As the extract filters through the funnel, collect the extracts for BNA, PCB, Pesticide, and TPH analysis in a KD apparatus; and collect the extracts for PAH analysis in a Turbovap tube. Low volume extracts are collected in 25 mL condensers. Rinse the sodium sulfate with additional methylene chloride.

12.14 Add an additional aliquot of dichloromethane to the separatory funnel and repeat the operations in Sections 12.9 to 12.11 twice, for a total of three extractions. After second and third addition of the extract to the sodium sulfate funnel, rinse the extract with an additional 6 mLs of methylene chloride.

12.15 For samples to be analyzed by Method 8270C and the target analyte list includes basic compounds, adjust the aliquot to $\text{pH} \geq 11$ with 10 N sodium hydroxide solution and repeat the operations in Sections 12.9 to 12.12, combining all extracts in the same concentrator tube.

12.16 Concentration

12.16.1 Low volume concentration for PAH analysis:

12.16.1.1 Add 1 boiling chip to each condenser and attach an open end Snyder column using cut-resistant gloves.

12.16.1.2 Place the K-D apparatus on the water bath with the receiver tube partially immersed in the water. The temperature of the water bath is 65-70°C.

12.16.1.3 Remove the K-D apparatus from the water batch when the extract volume is approximately 0.5 mL. After the unit has cooled, wearing cut-resistant gloves, remove the Snyder column. Proceed to Section 12.18 Finalizing to complete the quantitative transfer.

12.16.2 Kuderna-Danish Method

12.16.2.1 Add 1-3 boiling chips to each flask and attach a Snyder column

12.16.2.2 Place the apparatus on the water bath with the receiver tube partially immersed in the water. The temperature of the water bath is 65-70°C. The balls in the Snyder column should actively chatter, but not flood with solvent through the concentration process.

12.16.2.3 Remove the K-D from the water bath when the extract volume is 3-5 mL. After the unit has cooled, remove the Snyder column. Rinse the flask with dichloromethane before removing the Receiver tube. Proceed to Section 12.18 Finalizing to complete the quantitative transfer.

12.16.3 Turbo Vap® Method (Dry-Vap)

12.16.3.1 Place the concentrator receiver into the evaporator unit. Cover with a piece of aluminum foil held on with a rubber band to avoid cross contamination. A disposable pipette is used to create a hole in the aluminum foil to allow the solvent to escape.

12.16.3.2 Operate the unit according to the manufacturer's instructions until the extract volume is 1-5 mL. Remove the receiver and allow to cool. Rinse down the sides of the flask.

12.17 Solvent – Exchange – Extracts to be analyzed by Methods 8081A and 8082 must be exchanged to hexane.

12.18 Finalizing

12.18.1 Further concentrate extracts and the concentrator rinses to approximately ½ of the target final volume by a gentle stream of dry nitrogen using a blow down manifold or N-EVAP. BNA final volume is then archived by putting on hot plate water bath. Samples are brought up to 1 mL final volume in a syringe. PAH Extracts concentrated by the Turbo Vap® method are brought to final volume in the Turbo Vap tube. Extracts can also be concentrated by adding a fresh boiling chip, fitting the K-D receiver tube with a micro Snyder column, and returning the tubes to the heated water bath.

12.18.2 Quantitatively transfer the target final extract volume of 1.0 mL to a 2.0-mL vial for PAH and BNA analyses. Quantitatively transfer the target final extract volume of 1 mL to a 1 mL vial for TPH analyses. Quantitatively transfer the target final extract volume of 10 mL to a 10-mL vial for Pesticides, PCB, and Toxaphene analyses. Final extract volumes may be adjusted to meet client requirements.

12.18.3 Dilute the final extract to volume with the final solvent. The TPH extract is ready for analysis without diluting to volume.

12.18.4 If the final extract will not be analyzed immediately, it must be stored at $\leq -10^{\circ}\text{C}$ or at $\leq 6^{\circ}\text{C}$, as required by the SOP for the determinative procedure.

12.18.5 If the extract cannot be concentrated to the volumes specified above, it should be diluted or subjected to an approved extract cleanup procedure.

13 Quality Control

13.1 Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples or 12-hour window (whichever is most frequent)	Target analytes must be less than reporting limits	Re-extract and re-analyze associated samples if blank result is greater than RL
Laboratory Control Sample (LCS)	All target analytes	One per batch of up to 20 samples	See analytical SOPs	Re-extract and re-analyze associated samples if LCS is outside acceptance criteria were possible. Exceptions: 1) If LCS recovery is > QC limits and target analytes are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)/Matrix Spike Duplicate	All target analytes	One set per batch of up to 20 samples	See analytical SOPs	No corrective actions necessary. If LCS recovery is in range, the system is considered valid and the out-of-control MS/MSDs are footnoted appropriately by the analyst.
Surrogates	All applicable surrogate compounds	Added to each sample, blank and QC sample	See analytical SOPs	Surrogates above limits but no hits-report samples with footnote. Surrogate limits above limits but with hits- re-extract if possible or report as biased high. Surrogates below limits: re-extract if possible or report as biased low.

14 Data Analysis and Calculations

14.1 Refer to Pace SOP: S-GB-Q-009 *Common Laboratory Calculations* (most current revision or replacement) to calculate standard concentrations as required.

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 Refer to Table 13.1

16 Corrective Actions for Out-of-Control Data

16.1 Refer to Table 13.1

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 Refer to Table 13.1

18 Method Performance

- 18.1 Method Detection Limit (MDL) Study:** An MDL study must be conducted every 12 months per S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement) for each matrix per instrument.
- 18.2 Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement).
- 18.3** Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, most current revision or replacement, to demonstrate continuing competence. All results are stored in the quality assurance office.

19 Method Modifications

- 19.1** Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure
- 19.2** All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- 19.3** Procedures identified as “Best Practices” by PACE 3P Programs will be incorporated into this document as minimum requirements for Pace Laboratories.
- 19.4** If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.

20 Instrument/Equipment Maintenance

- 20.1** Refer to the most current revision or replacement of S-GB-O-030, *Support Equipment*.

21 Troubleshooting

- 21.1** Not applicable to this SOP.

22 Safety

- 22.1 Standards and Reagents:** The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.1.1 Methylene chloride creates excessive pressure very rapidly when shaken in an enclosed apparatus, as directed in this method. The shaker funnels used in conjunction with the extraction vessel are designed to automatically vent excess pressure created by methylene chloride. As a result, the extraction mixer should be operated in a hood to avoid exposure of the technician to solvent vapors.

22.2 Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

23 Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24 Pollution Prevention and Waste Management

24.1 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25 References

25.1 “Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”; EPA SW-846, latest revision. Method 3510C “Separatory Funnel Extraction”.

25.2 Pace Analytical Quality Manual; latest revision.

25.3 The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

25.4 Pace SOP: S-GB-I-067, *TCLP, SPLP, and ASTM Procedures* (most current revision or replacement)

25.5 Pace SOP: S-GB-O-049, *Determination of Semi-Volatile Organics by GC/MS* (most current revision or replacement)

25.6 Pace SOP: S-GB-O-050, *Determination of Semi-Volatile Organics by GC/MS (Selective Ion Monitoring)* (most current revision or replacement)

25.7 Pace SOP: S-GB-O-026, *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography* (most current revision or replacement)

25.8 Pace SOP: S-GB-O-027, *Analysis of Organochlorine Pesticides by Gas Chromatography* (most current revision or replacement).

25.9 Pace SOP: S-GB-O-028, *Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes* (most current revision or replacement).

25.10 Pace SOP: S-GB-O-032, *Gel Permeation Chromatography* (most current revision or replacement).

25.11 Pace SOP: S-GB-O-034, *Sulfuric Acid Cleanup* (most current revision or replacement)

25.12 Pace SOP: S-GB-O-015, *Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics* (most current revision or replacement).

25.13 Pace SOP: S-GB-O-023, *Total Petroleum Hydrocarbons* (most current revision or replacement).

26 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1 Not applicable to this SOP.

27 Revisions

Document Number	Reason for Change	Date
SOT-ALL-O-003-rev.00	Restructured document format to new corporate template. Updated formatting and Responsibilities and Distribution section	16Nov2006
S-ALL-GB-O-003-Rev.00	First issue as Corporate Template	10Jun2008
S-GB-O-053-Rev.00	Updated cover page to Periodic Review Renamed SOP to be consistent with SOP: S-ALL-Q-003 <i>Document Numbering</i> Section 7.0: Included TCLP/SPLP Preservation/Hold time Section 11.4: Added spiking/surrogate solutions must be added to sample prior to adding Methylene Chloride Section 11.4.1 and 11.4.2 Included TCLP/SPLP Extraction Procedure information. Updated table in section 8.1, 8.2, 9.1, 9.3, and 9.4 Added TPH standards and procedures	04Jan2010
S-GB-O-053-Rev.01	Updated SOP reference S-ALL-GB-O-001 to S-GB-O-049. Updated SOP reference S-ALL-GB-O-008 to S-GB-O-050 Section 13.1: Updated SOP to S-GB-Q-020: Determination of the LOD and LOQ.	07Jul2011
S-GB-O-053-Rev.02	Throughout Document: Updated formatting and font. Section 2.1: Updated Summary to include 100mL container volume. Table 7.1: Added separate statement for PCB hold time to a 365 day extraction and 365 analytical hold time. Added additional row to address Aqueous PAH Low Volume extraction container, preservation and hold time. Table 8.2: General Supplies: Added 125mL Separatory Funnels, 25mL Condenser Tubes, and Snyder Columns. Table 9.3, 9.4 and 9.5: Added standard solutions for PAH Low Volume Extractions. Section 11: Added PAH Low Volume Procedure throughout section. Section 11.16.1: Added Low Volume PAH Concentration Procedure.	02May2012

Document Number	Reason for Change	Date
S-GB-O-053-Rev.03	Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 <i>Preparation of SOPs</i> Throughout Document: Renamed Tables to be consistent with current Section; Updated internal section references; Updated SOP references. Table 7.1: Updated to 14 day hold time for tumbling and changed the extraction hold time starts when the tumbling time starts. Section 12.8: Updated pH range for 8081/8082 is 5-9, not 11. Table 13.1: Changed LCS corrective action to reextract all samples and associated QC if outside of acceptance criteria.	26Jun2014

ATTACHMENT B2



Pace Analytical Services, Inc
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469 2436
Fax: 920 469 8827

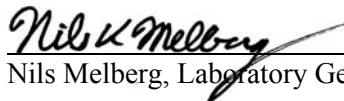
STANDARD OPERATING PROCEDURE

DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS AND SIM (SELECTIVE ION MONITORING)

Reference Methods: EPA SW-846 Method 8270C SIM

Local SOP Number:	S-GB-O-050-Rev.03
Effective Date:	Date of Final Signature
Supersedes:	S-GB-O-050-Rev.02
SOP Template Number:	SOT-ALL-O-008-rev.01

APPROVALS


Nils Melberg, Laboratory General Manager

01/27/14
Date


Kate Grams, Laboratory Quality Manager

1/27/14
Date


Chris Haase, Laboratory Department Manager

1/27/14
Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Green Bay to determine the concentration of Semi-volatile Organic Compounds in environmental samples using Selective Ion Monitoring (SIM). The laboratory utilizes GC/MS and bases these documented procedures on those listed in EPA SW-846 Method 8270C SIM. Samples for analysis are prepared by SW846 Method 3510C and SW846 Method 3546 following Pace SOPs S-GB-O-053, *Separatory Funnel Extraction by SW846 3510C* (most current revision or replacement), and S-GB-O-045, *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons and Base/Neutral/Acid, and Total Petroleum Hydrocarbons in Solid Matrices by SW846 3546* (most current revision or replacement).

2. Summary of Method

2.1. Sample extracts are prepared for analysis by an appropriate sample preparation method. The semivolatile organic compounds are introduced into the gas chromatograph (GC) by injecting an aliquot of the sample extract. The GC conditions are programmed to separate the analytes. The GC effluent is directly introduced to a mass spectrometer (MS) for both identification and quantification of analytes. Analytes are identified by comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

3. Scope and Application

3.1. This procedure is principally used to determine concentrations of polynuclear aromatic hydrocarbons (PAHs) but can also be used to determine concentrations of other neutral, acidic, and basic semi-volatile organic compounds in extracts prepared from many types of water samples, soil samples and wastes. Analytes must be soluble in dichloromethane and amenable to capillary gas chromatography. A list of applicable compounds is shown herein in Table 11.1. Pace Reporting Levels (PRLs) are also shown for water and soil samples. PRLs are subject to change based on current analytical system performance and actual sample matrices.

3.2. This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of semi-volatile configured GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.4. This method cannot be substituted for other similar published methods where permit or regulatory compliance is required

4. Applicable Matrices

- 4.1. This SOP applies to surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, soils, and sludges.

5. Limits of Detection and Quantitation

- 5.1. The reporting limit (LOQ) for all analytes is listed in Table 11.1 through 11.3 for the listed matrices. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

- 6.1. Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

- 6.2. Matrix interferences may result from materials co-extracted from some samples.

- 6.3. Significant phthalate contamination may result at any time if consistent quality control is not practiced. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

- 6.4. Contamination by carryover can occur when high concentration extracts are analyzed prior to low concentration extracts. The contamination may also cause degradation of labile analytes. Whenever carryover is suspected, the affected extracts should be re-analyzed. If significant degradation of the GC/MS systems is suspected, system performances samples should be analyzed and corrective action taken as needed.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Storage, and Hold time

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One 1L amber glass	None	4 ± 2°C	7 days
HVI Aqueous	One 125 amber glass	None	4 ± 2°C	7 days
TCLP	One 1L amber glass	None	4 ± 2°C	TCLP Leachates must be solvent extracted within 7 days of the completion of the tumbling process
Soil/Solid (non-aqueous)	One 8oz wide glass jar	None	4 ± 2°C	14 days
Extracts	ONE 2mL glass vials, same as used for standard storage	None	≤ -10°C	40 days

8. Definitions

Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary.

- 8.1. Run Sequence Log—A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

- 8.2. Tune Period—The period after the DFTPP instrument tune check within which analyses may be performed.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 - Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatograph	HP	6890	40MSS2
Mass Selective Detector	HP	5973	40MSS2
Data System	HP	Chem Station	40MSS2
Auto-injector	HP	7683	40MSS2
Vacuum Pump (Rough)	HP	E2M2	40MSS2
Tray	HP	G2614A	40MSS2
Gas Chromatograph	HP	6890	40MSS3
Mass Selective Detector	HP	5973	40MSS3
Auto-injector	HP	7683	40MSS3
Tray	HP	G2614A	40MSS3
Vacuum Pump (Rough)	HP	G1099-80023	40MSS3
Data System	HP	Chem Station	40MSS3
Gas Chromatograph	HP	6890	40MSS4
Mass Selective Detector	HP	5973	40MSS4
Auto-injector	HP	7683	40MSS4
Tray	HP	G2614A	40MSS4
Gerstel Unit		MACH	40MSS4
Vacuum Pump (Rough)	HP	G1099-80023	40MSS4
Data System	HP	Chem Station	40MSS4
Gas Chromatograph	HP	6890	40MSS7
Mass Selective Detector	HP	5975	40MSS7
Auto-injector	HP	7683	40MSS7
Tray	HP	G2614A	40MSS7
Gerstel Unit		HVI	40MSS7
Vacuum Pump (Rough)	HP	G1099-80023	40MSS7
Data System	HP	Chem Station. Gerstel Maestro	40MSS7
Gas Chromatograph	HP	7890A	40MSS8
Mass Selective Detector	HP	5975C	40MSS8
Auto-injector	HP	7683	40MSS8
Tray	HP	G2614A	40MSS8
Agilent MMI		HVI	40MSS8
Vacuum Pump (Rough)	HP	G1099-80023	40MSS8
Data System	HP	Chem Station.	40MSS8

9.2. Table 9.2 - Chromatography Supplies

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	XTI-5	1223-124	30 m, 0.25 mm ID, 0.25 df
Fluorocarbon O-rings	Restek		20377	
Vespel/Graphite	Restek		20229	1/16" x0.4 mm ID
Gooseneck Splitless	Restek		20800	4 mm x 6.5 x 78.5 for
Guard Column	Restek		20231	0.5 MID
Ferrules	Fisher Scientific		5-5	
HVI items				
Analytical Column	Restek	XTI-5	1223-124	30 m, 0.25 mm ID, 0.25 df
Graphpack 3D ferrule	Gerstel		007541-005-00	Ferrule for glass inlet liner
Graphpack 2M Ferrules	Gerstel		001805-045-00	0.45mm ferrule
Glass wool filled Deactivated liner	Gerstel		010850-010-00	Inlet liner
Needle guide	Gerstel		003091-064-00	0.64mm Teflon needle
Vespel/Graphite	Restek		20229	1/16" x0.4 mm ID

9.3. Table 9.3 - Glassware

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	10mL, 25mL, 50mL	Class A
Glass Storage Vials	5mL, 10mL, 12mL, with Teflon-lined screw caps	MG Scientific/T102-3-INV, T102-1-INV
Glass Autosampler Vials	2.0mL with Teflon-lined crimp or screw caps	MG Scientific/V300-3/V300-20N
Borosilicate Inserts	200uL low volume vial inserts	

9.4. Table 9.4 - General Supplies

Supply	Description	Vendor/ Item # / Description
Gas tight syringes	10-uL, 25-uL, 50-uL, 100-uL, 250-uL, 500-uL, and 1000-uL, as needed, Hamilton or equivalent.	Fisher Scientific/Various
Teflon dispensing bottles		
Pipettes	Borosilicate Glass 9" Pipette	MG Scientific/D200-9

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Stock Standards

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Dichloromethane		Pesticide Grade or equivalent

Table 10.2 - Standard Definitions

Standard	Description	Comments
Tune Standard	Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine solution in dichloromethane used to verify ion response ratios and system inertness prior to analysis. [For PAH only analysis, breakdown and tailing factors do not need to be evaluated or controlled.]	Must inject no more than 50ng on column. The DFTPP must meet ion ratio criteria as per Section 12.2.1 [Some programs may not require that DFTPP meets ion ratio criteria. Labs must minimally analyze a DFTPP standard to verify mass axis alignment.]
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Naphthalene-d ₈ Acenaphthene-d ₁₀ Phenanthrene-d ₁₀ Chrysene-d ₁₂ Perylene-d ₁₂
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to analysis.	2-Fluorobiphenyl Terphenyl-d ₁₄
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	

10.2. Table 10.3 - Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	<ul style="list-style-type: none"> Concentrated reference solution purchased directly from approved vendor 	<ul style="list-style-type: none"> Manufacturer's recommended expiration date for unopened ampulated standards. Stock standards must be replaced 1 year after ampule is opened or on expiration date, whichever is sooner. 	<ul style="list-style-type: none"> Manufacturer's recommended storage conditions When standard is opened, record all information in the standard prep log program in Epic Pro.
Intermediate and Working Standard Solutions	<ul style="list-style-type: none"> Reference solutions prepared by dilutions of the stock solution 	<ul style="list-style-type: none"> 1 year from preparation or the expiration date listed for the stock source, whichever is sooner. Working solutions must be checked frequently and replaced if degradation or evaporation is suspected. 	<ul style="list-style-type: none"> Store in amber vials with Teflon lined screw caps Manufacturer's recommended storage conditions for stock source solution. If stock source conditions conflict, store according to method requirements.

10.3. Standard Sources: Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials, recipes for preparing dilutions and working standards, and concentrations for all compounds are presented in table 10.5. All intermediate standards are prepared using dichloromethane and stored in glass vials with Teflon lined caps or Mininert valves or as recommended by the standard manufacturer.

10.4. Stock Standards:

10.4.1. Table 10.5 Stock Standards

Standard	Conc.	Purity	Manufacturer	Vendor	Catalog #
DFTPP Tuning Standard	1000 ug/mL	99%	Supelco	Supelco	47548-4
Internal Standard	4000 ug/mL	99%	Restek	Restek	31006
Surrogate Standard	5000 ug/mL	99%	Restek	Restek	31082
Calibration Standard	500 ug/mL	99%	Accustandard	Accustandard	M-610-FL-R-5X
Calibration Standard	500 ug/mL	99%	O2SI	O2SI	114132-05-5pk
Benzo(e)pyrene Calibration Standard	1000 ug/mL	99%	Absolute Standard	Absolute Standard	71016
Initial Calibration Verification Standard	2000 ug/mL	99%	Supelco	Supelco	47543-U
Initial Calibration Verification Standard for Benzo(e)pyrene	50ug/mL	99%	Accustandard	Accustandard	H-112S

10.5. Working Standard Preparation: Working calibration standards are prepared in dichloromethane or a water soluble solvent. Standards made for direct analysis on the GC/MS are made in dichloromethane. Standards made for addition into samples as part of the preparation are made into dichloromethane. Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks or prepared in smaller, glass vials and brought to volume by additions of solvent with micro syringes.

10.5.1. Volumetric Flask Preparation – Fill appropriate volumetric flask 2/3 full with dichloromethane. Introduce appropriate amount of standard into dichloromethane in flask with a micro-syringe. Dilute to volume with dichloromethane.

10.6. Calibration Standard Preparation: Calibration standards are made into dichloromethane for the purpose of direct analysis by the analytical instrumentation. The standards must be made in a volumetric fashion. Several alternatives exist but the method employed by Pace – Green Bay utilizes volumetric flasks according to the following procedure.

10.6.1 Standards are prepared from commercially available multi-component stock solutions. The sources of materials, recipes for preparing dilutions and working standards, and concentrations for all compounds are presented below. All standards are prepared using dichloromethane and stored in clear vials with PTFE lined caps.

10.6.2 Storage and Stability of Analytical Standards –All standards must be stored in the dark at less than -10°C or at a temperature recommended by the manufacturer. They must be replaced every 12 months or sooner if the standards show signs of degradation. As each standard, from the vendor is opened, record all pertinent information in the electronic standards logbook in Epic Pro. Record all standard preparations in the standard logbook.

10.7. Preparation Procedures

10.7.1. DFTPP Tuning Solution:

10.7.1.1 **Soil and Aqueous Analysis:** A Dichloromethane solution containing 50ng/uL of decafluorotriphenylphosphine (DFTPP) is prepared. A stock solution; Supelco cat.# 47548-4; containing DFTPP at 1000ug/mL, is diluted taking 1.25mL and diluting to 25mL of dichloromethane.

10.7.1.2 **Aqueous High Volume Injection:** A Dichloromethane solution containing 1ng/uL of decafluorotriphenylphosphine (DFTPP) is prepared. The Soil and Aqueous Intermediate Tune Solution from Section 10.7.1.1; containing DFTPP at 50ug/mL, is diluted taking 20.0uL and diluting to 1mL of dichloromethane.

10.7.2. Internal Standard Solutions:

10.7.2.1 **Stock Solution** – Obtained from Restek cat. #31006 containing 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12 and Perylene-d12 all at 4000ug/mL in dichloromethane.

10.7.2.1.1 **Water Intermediate Standard** - This intermediate standard is prepared by diluting 50uL up to 10mLs of dichloromethane, producing a solution that is 20ug/mL for each component. 10uL of this standard is added to 1mL of every standard, sample, spike and blank analyzed by this method. Producing an on-column concentration of 200ug/L.

10.7.2.1.2 **Water Intermediate HVI Standard** - This intermediate standard is prepared by diluting 25uL up to 50mLs of dichloromethane, producing a solution that is 4ug/mL for each component. 10uL of this standard is added to 1mL of every standard, sample, spike and blank analyzed by this method. Producing an on-column concentration of 40ug/L.

10.7.2.1.3 **Soil Intermediate Standard** - This intermediate standard is prepared by diluting 500uL up to 1.0mL of dichloromethane, producing a solution that is 2000 ug/mL for each component. 10uL of this standard is added to 1mL of every standard, sample, spike and blank analyzed by this method. Producing an on column concentration of 20ug/mL.

10.7.3. Surrogate Standard Solution:

10.7.3.1 **Water Working Standard:** Water Stock Solution obtained from Restek, cat. #31082 containing 1,2-Dichlorobenzene-d4, Nitrobenzene-d5, 2-Fluorobiphenyl and p-Terphenyl-d14 all at 1000ug/mL in dichloromethane. The working standard is prepared by diluting 100uL up to 500mLs of dichloromethane, producing a standard that is 0.2ug/mL of each component. 1.0mL of this working standard is spiked into all QC and Samples, producing and on-column value of 200ug/L.

10.7.3.2 **Water HVI Working Standard:** Water Stock Solution obtained from Restek, cat. #31082 containing 1,2-Dichlorobenzene-d4, Nitrobenzene-d5, 2-Fluorobiphenyl and p-Terphenyl-d14 all at 1000ug/mL in dichloromethane. The working standard is prepared by diluting 100uL up to 500mLs of dichloromethane, producing a standard that is 0.2ug/mL of each component. 100uL of this working standard is spiked into all QC and Samples, producing and on-column value of 200ug/L.

10.7.3.3 Soil Working Standard: Soil Stock Solution obtained from Restek. Cat #31082 containing 100uL 1,2-Dichlorobenzene-d₄, Nitrobenzene-d₅, 2-Fluorobiphenyl and p-Terphenyl-d₁₄ all at 5000ug/mL in dichloromethane. The working standard is prepared by diluting 3000uL up to 100mLs of dichloromethane, producing a standard that is 150ug/mL of each component. 100uL of this working standard is spiked into all QC and Samples, producing an on-column value of 15ug/mL.

10.7.4. Laboratory Control Spike/Matrix Spike/Duplicate Solution:

10.7.4.1 Water LCS/MS Standard: Water Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. Intermediate standard is prepared by diluting 200uL of the 500ug/mL up to 200mLs with Dichloromethane. 1.0mL of this standard is spiked into all Lab Control and Matrix Spikes, producing an on-column concentration of 200ug/L.

10.7.4.2 Water LCS/MS HVI Standard: Water Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. Intermediate standard is prepared by diluting 200uL of the 500ug/mL up to 200mLs with Dichloromethane. 100uL of this standard is spiked into all Lab Control and Matrix Spikes, producing an on-column concentration of 200ug/L.

10.7.4.3 Soil Standard: Stock solution is obtained from O2SI, product # 114132-05-5pk, containing the 18 PAH compounds, all at 500ug/mL. 20 uL of this solution is spiked into all LCS and MS samples, producing an on-column concentration of 10ug/mL.

10.7.5. Initial Calibration Intermediate Standard

10.7.5.1 Water Samples: Stock solution is obtained from O2SI, product #114132-05-5pk, containing the 18 PAH compounds, all at 500ug/mL. The intermediate is prepared by diluting 20uL of the 500ug/L PAH stock along with 67uL of the 150ug/L surrogate intermediate (9.7.2.2.3) up to 10.0mLs dichloromethane, producing an intermediate calibration standard that is 1000ug/L of all components.

10.7.5.2 HVI Water Samples: Stock solution is obtained from O2SI, product #114132-05-5pk, containing the 18 PAH compounds, all at 500ug/mL. The intermediate is prepared by diluting 20uL of the 500ug/L PAH stock along with 67uL of the 150ug/L surrogate intermediate (9.7.2.2.3) up to 100mLs dichloromethane, producing an intermediate calibration standard that is 100ug/L of all components.

10.7.5.3 Soil Samples: Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. The intermediate is prepared by diluting 500 uL of the 500 ug/mL PAH Stock Standard along with 50 uL of the 5000 ug/mL B/N Surrogate Stock Solution to 10.0 mL with methylene chloride producing the high standard of 25 ug/mL. Per Client Request Benzo(e)pyrene may be included as a separate calibration curve. Stock Solution is obtained from Absolute Standards, catalog # 71016, containing Benzo(e)pyrene at 1000ug/mL. The intermediate is prepared by diluting 250uL of 1000ug/mL Benzo(e)pyrene Stock Standard up to 10.0mLs dichloromethane, producing an intermediate calibration standard that is 25ug/mL. The standards are then further diluted to obtain the concentrations used for calibration.

10.7.6. Initial Calibration Working Standards:

10.7.6.1 **Water Initial Calibration Curve:** The following working calibration standards are prepared by using the Initial Calibration Intermediate Standard for Water Samples listed above (10.7.5.1):

Standard Concentration (ng on column)	Amount of Intermediate Standard Added (uL)	Amount of Intermediate Internal Standard Added (uL)	Final Volume (uL)
25	25	10	1010
50	50	10	1010
100	100	10	1010
200	200	10	1010
300	300	10	1010
500	500	10	1010
1000	1000	10	1010
CCV - 200	200	10	1010

10.7.6.2 **HVI Water Initial Calibration Curve:** The following working calibration standards are prepared by using the Initial Calibration Intermediate Standard for Water Samples listed above (10.7.5.2)

Standard Concentration (ng on column)	Amount of Intermediate Standard Added (uL)	Amount of Intermediate Internal Standard Added (uL)	Final Volume (uL)
1.0	10	10	1010
2.5	25	10	1010
5.0	50	10	1010
10.0	100	10	1010
20.0	200	10	1010
30.0	300	10	1010
50.0	500	10	1010
100.0	1000	10	1010
CCV – 20.0	200	10	1010

10.7.6.3 Soil Initial Calibration Curve: The following working calibration standards are prepared by using the Initial Calibration Intermediate Standard for Soil Samples listed above (10.7.5.3):

Standard Concentration (ng on column)	Amount of Intermediate Standard Added (uL)	Amount of Intermediate Internal Standard Added (ul)	Final Volume (uL)
0.25	10	10	1010
0.50	20	10	1010
1.0	40	10	1010
4.0	160	10	1010
10.0	400	10	1010
20.0	800	10	1010
25.0	1000	10	1010
CCV – 10.0	400	10	1010

10.7.7. Initial Calibration Verification Standards:

10.7.7.1 Water Initial Calibration Verification Standard: Stock Solution is obtained from Supelco, cat# 47543-U, containing the 18 PAH compounds all at 2000ug/mL. A working solution is prepared by diluting 5uL of the 2000ug/mL PAH stock, along with 67uL of the 150ug/mL B/N surrogate intermediate (9.7.3.3) up to 50mLs with dichloromethane. This produces an ICV with a concentration of 200ug/L.

10.7.7.2 HVI Water Initial Calibration Verification Standard: 100 uL Intermediate Solution 9.7.7.1 containing the 18 PAH compounds all at 200ug/mL is diluted up to 1mL with dichloromethane. This produces an ICV with a concentration of 20.0ug/L.

10.7.7.3 Soil Initial Calibration Verification Standard: Stock solution is obtained from Supelco, cat # 47543-U, containing the 18 PAH compounds, all at 2000 ug/mL. An intermediate is prepared by diluting 100 uL of the 2000 ug/mL PAH Stock along with 40 uL of 5000 ug/mL B/N Surrogate Stock Solution to 2.0 mL with dichloromethane. To produce the working ICV solution, the above intermediate ICV solution is further diluted by taking 100 uL of the intermediate ICV and diluting to 1.0 mL with dichloromethane to produce a 10 ug/mL working ICV standard. A separate Benzo(e)pyrene ICV is required. Stock Solution is obtained from Accustandard, catalog # H-112S, containing Benzo(e)pyrene at 50ug/mL. The working ICV solution is prepared by diluting 200uL of 50ug/mL Benzo(e)pyrene Stock Standard up to 1.0mLs dichloromethane to produce a working initial calibration verification standard that is 10ug/L.

11. Calibration and Standardization

11.1. Tune Verification – The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. For analysis by Selective Ion Monitoring, the response ratio criteria for DFTPP may be immaterial unless a custom spectral library were established under SIM conditions. Therefore, unless a program specific requirement mandates ion ratio criteria be followed, the laboratory must analyze a DFTPP standard but only for the purpose of verifying the alignment of the mass spectral axis. If the quality program requires otherwise and the ion ratios do not meet the criteria for the method, follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures.

11.2. Initial Calibration:

11.2.1. Analysis of Standards: An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 11.1 for compound concentrations.

Table 11.1: Calibration standard compound concentrations for Water Analysis

Analyte	PQL water (ug/L)	Std 1 ug/m L	Std 2 ug/m L	Std 3 ug/m L	Std 4 ug/m L	Std 5 ug/m L	Std 6 ug/m L	Std 7 ug/m L
Acenaphthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Acenaphthylene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Anthracene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benz(a)anthracene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(a)pyrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(b)fluoranthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(g,h,i)perylene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(k)fluoranthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Chrysene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Dibenz(a,h)anthracene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Fluoranthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Fluorene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Indeno(1,2,3-cd)pyrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
2-Methylnaphthalene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Naphthalene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Phenanthrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Pyrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
1-Methylnaphthalene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0

Table 11.2: Calibration standard compound concentrations for HVI Water Analysis

Analyte	PQL water (ug/L)	Std 1 ug/m L	Std 2 ug/mL	Std 3 ug/m L	Std 4 ug/ mL	Std 5 ug/m L	Std 6 ug/m L	Std 7 ug/m L	Std 8 ug/m L
Acenaphthene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Acenaphthylene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Anthracene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Benz(a)anthracene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Benzo(a)pyrene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Benzo(b)fluoranthene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Benzo(g,h,i)perylene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Benzo(k)fluoranthene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Chrysene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Dibenz(a,h)anthracene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Fluoranthene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Fluorene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Indeno(1,2,3-cd)pyrene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
2-Methylnaphthalene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Naphthalene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Phenanthrene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
Pyrene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1
1-Methylnaphthalene	0.050	0.001	0.0025	0.005	0.01	0.02	0.03	0.05	0.1

Table 11.3: Calibration standard compound concentrations for Soil Analysis

Analyte	PQL soil (ug/kg)	Std 1 ug/L	Std 2 ug/L	Std 3 ug/L	Std 4 ug/L	Std 5 ug/L	Std 6 ug/L	Std 7 ug/L
Acenaphthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Acenaphthylene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Anthracene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benz(a)anthracene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(a)pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(b)fluoranthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(g,h,i)perylene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(k)fluoranthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Chrysene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Dibenz(a,h)anthracene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Fluoranthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Fluorene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Indeno(1,2,3-cd)pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
2-Methylnaphthalene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Naphthalene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Phenanthrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
1-Methylnaphthalene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3

11.2.2. Calibration Response Factors: Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

11.2.3. To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard ($\mu\text{g/L}$).

C_x = Concentration of the compound being measured ($\mu\text{g/L}$).

11.2.4. Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8270C method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples. For the SIM method, all target compounds are considered to be SPCC compounds and the average RF for each SPCC compound must be as follows:

Naphthalene	≥ 0.700
2-Methylnaphthalene	≥ 0.400
Acenaphthylene	≥ 0.900
Acenaphthene	≥ 0.900
Fluorene	≥ 0.900
Phenanthrene	≥ 0.700
Anthracene	≥ 0.700
Fluoranthene	≥ 0.600
Pyrene	≥ 0.600
Benzo(a)anthracene	≥ 0.800
Chrysene	≥ 0.700
Benzo(b)fluoranthene	≥ 0.700
Benzo(k)fluoranthene	≥ 0.700
Benzo(a)pyrene	≥ 0.700
Benzo(e)pyrene	≥ 0.700
Indeno(1,2,3-cd)pyrene	≥ 0.500
Dibenzo(a,h)anthracene	≥ 0.400
Benzo(g,h,i)perylene	≥ 0.500

Two of the PAH's response factors are allowed to not meet these requirements.

11.2.5. Calibration Curve Fit: The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the “goodness of fit”.

11.2.6. Average Response Factor (ARF): The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF:

$$\%RSD = SD * 100 / ARF$$

Where: SD = Standard deviation of the averaged RFs for a given compound

11.2.7. The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The **Calibration Check Compounds (CCCs)** are a subset of the target analyte list that must meet specific criteria for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria ($\leq 30\%$ for CCCs). If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated. **For the SIM method, all target compounds are considered CCC compounds.**

11.2.8. Linear Regression: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y=ax+b$ where “a” is the slope of the line and “b” is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient “r” is used to determine the acceptability of a linear regressed curve.

11.2.9. Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

11.2.10. A calculation of the coefficient of determination (COD) is used to determine the acceptability of a non-linear regressed curve. Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious

problem is discovered and documented. The point must be dropped in its entirety and reanalyzed. Re-analysis should be within the same 12 hour time window and must occur within 8 hours of the original analysis.

11.3. Calibration Verification:

11.3.1. **Second Source Verification:** In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the “fit” of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

11.3.2. Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = \frac{(RF_{ccv} - AveRF_{cal})}{AveRF_{cal}} * 100$$

$$\% \text{ Drift} = \frac{(\text{Result CCV} - \text{True Value CCV})}{\text{True Value CCV}} * 100$$

11.3.3. **Continuing Calibration Verification (CCV):** As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished by analyzing a midpoint calibration standard (CCV).

11.3.4. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

11.3.5. The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\% \text{ Difference} = \frac{(RF_{ccv} - AveRF_{cal})}{AveRF_{cal}} * 100$$

$$\% \text{ Drift} = \frac{(\text{Result CCV} - \text{True Value CCV})}{\text{True Value CCV}} * 100$$

Table 11.4: Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD \leq 15%	If not met, try linear regression fit
	Linear Regression	$r \geq 0.99$	
	Non-linear Regression	COD ≥ 0.99	If not met, try non-linear regression fit
			If not met, remake standards and recalibrate
System Performance Check Compounds (SPCCs)	All target compounds	Naphthalene ≥ 0.700 2-Methylnaphthalene ≥ 0.400 Acenaphthylene ≥ 0.900 Acenaphthene ≥ 0.900 Fluorene ≥ 0.900 Phenanthrene ≥ 0.700 Anthracene ≥ 0.700 Fluoranthene ≥ 0.600 Pyrene ≥ 0.600 Benzo(a)anthracene ≥ 0.800 Chrysene ≥ 0.700 Benzo(b)fluoranthene ≥ 0.700 Benzo(k)fluoranthene ≥ 0.700 Benzo(a)pyrene ≥ 0.700 Benzo(e)pyrene ≥ 0.700 Indeno(1,2,3-cd)pyrene ≥ 0.500 Dibenzo(a,h)anthracene ≥ 0.400 Benzo(g,h,i)perylene ≥ 0.500 Two of the PAH's response factors are allowed to not meet these requirements.	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system. Additional client specific requirements for the analysis of contract samples requires that all PAH's and surrogate compounds also be considered SPCCs and must meet the minimum RRF criterion of 0.05.
Calibration Check Compounds (CCCs)	All target compounds	%RSD $< 30\%$	%RSD for the calibration check compounds (CCC's) must be $\leq 30\%$ regardless of curve fit used.
Second Source Verification Standard	Immediately after each initial calibration	% Drift $\pm 30\%$ % Diff $\pm 30\%$	Acceptance criteria are $\pm 30\%$ for all analytes. Additional client specific requirements for the analysis of contract samples requires that all compounds must be within $\pm 20\%$
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT	RT ± 30 sec	Use midpoint calibration standard as reference
	Internal Standard Response	50 – 200%	Use midpoint calibration standard as reference
	CCCs	RF $\pm 20\%$ Diff. Result $\pm 20\%$ Drift	Use for Avg RF calibration curves
			Use for linear and non-linear calibration curves

11.4. Calibration Corrective Actions:

11.4.1. Calibration Linearity Problems:

- 11.4.1.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.
- 11.4.1.2 Perform another initial calibration.
- 11.4.1.3 No data can be reported
- 11.4.1.4 Generate a Non-Conformance Memo.

11.4.2. Second Source Verification Problems:

- 11.4.2.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.
- 11.4.2.2 Perform another initial calibration.
- 11.4.2.3 No data can be reported.
- 11.4.2.4 Generate a Non-Conformance Memo.

11.4.3. Continuing Calibration Verification Problems:

- 11.4.3.1 Reanalyze the original CCV standard to determine instrument consistency.
- 11.4.3.2 Prepare and analyze a new CCV standard to determine preparation consistency/standard integrity.
- 11.4.3.3 Document instrument maintenance.
- 11.4.3.4 Reanalyze CCV standard to determine if maintenance was effective in restoring performance. If the second CCV is acceptable, the analytical sequence is continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated.
- 11.4.3.5 Complete recalibration of instrument.
- 11.4.3.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the injection log with a thorough explanation for the deviation from policy.
- 11.4.3.7 Exceptions: If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

11.4.4. General Comment: When constructing a linear initial calibration curve, the analyst can drop curve points as follows:

The lowest curve point can be dropped as long as there is a standard that can meet the necessary reporting limits of the associated samples (or the reporting limits would have to be raised accordingly).

The highest curve point, or points, can be dropped but this decreases the upper calibration range, thereby limiting the analyst to reporting data within this new calibration range (this may cause more dilutions).

Mid-point injections in the curve can be removed if it can be proven that it was a bad injection, instrument failure, etc. The same curve point is required to be reanalyzed and incorporated in the curve prior to sample analysis. A supervisor or the Quality Manager is required to approve any such instance.

12. Procedure

12.1. Operating Parameters: Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 12.1: Instruments and Operating Parameters

Instrument IDs	Component	Settings and Consumables	
40MSS2	Gas Chromatograph	Column: Restek XTI-5 w/ Integruguard 30m, 0.25mm ID, 0.25df Inlet Liner: Restek 4 mm Single Gooseneck Injection Port Liners Inlet Seal: Restek Gold Plated inlet seal Column Ferrules: Restek 0.4mm Vespel/Graphite	Pressure / Flow: 2.0mL/min Initial Temperature: 100°C Initial Time: 1.5 min Final Temperature: 60°C/min to 155°C 20°C/min to 320°C 3 min hold Final Time: 13.61 min Injector Temperature: 295°C Detector Temperature:
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	1.0 uL Injection 3 Solvent rinses from each solvent vial	
Instrument IDs	Component	Settings and Consumables	
40MSS3	Gas Chromatograph	Column: Restek XTI-5 w/ Integruguard 30m, 0.25mm ID, 0.25df Inlet Liner: Restek 4 mm Single Gooseneck Injection Port Liners Inlet Seal: Restek Gold Plated inlet seal Column Ferrules: Restek 0.4mm Vespel/Graphite	Pressure / Flow: 2.0mL/min Initial Temperature: 100°C Initial Time: 1.5 min Final Temperature: 60°C/min to 155°C 20°C/min to 320°C 3 min hold Final Time: 13.61 min Injector Temperature: 295°C Detector Temperature:
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	1.0 uL Injection 3 Solvent rinses from each solvent vial	
Instrument IDs	Component	Settings and Consumables	
40MSS4	Gas Chromatograph	Column: Restek XTI-5 w/ Integruguard 30m, 0.25mm ID, 0.25df Inlet Liner: Restek 4 mm Single Gooseneck Injection Port Liners Inlet Seal: Restek Gold Plated inlet seal Column Ferrules: Restek 0.4mm Vespel/Graphite	Pressure / Flow: 2.0mL/min Initial Temperature: 100°C Initial Time: 1.5 min Final Temperature: 60°C/min to 155°C 20°C/min to 320°C 3 min hold Final Time: 13.61 min Injector Temperature: 295°C Detector Temperature:
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	1.0 uL Injection 3 Solvent rinses from each solvent vial	

Instrument IDs	Component	Settings and Consumables	
40MSS7	Gas Chromatograph	Column: Restek XTI-5 w/ Integruguard 30m, 0.25mm ID, 0.25df Inlet Liner: Gerstel Injection Port Liners Gerstel 0.45mm graphpack 2M ferrule Column Ferrules: Restek 0.4mm Vespel/Graphite 0.64 Teflon Needle Guide Graphpack 3D inlet liner ferrule.	Mode: PTV Solvent Vent Pressure : 25PSI Total Flow: 55.5mL/min Initial Temperature: 100°C Initial Time: 2 min Final Temperature: 20°C/min to 340°C 1.5 min hold Final Time: 15.5 min Initial Injector Temperature: 30°C Initial Time: 1.3 min Final Temperature: 12°C/s to 300°C 13.5 min hold Detector Temperature: 300
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	50.0 uL Injection 3 Solvent rinses from each solvent vial	

12.2. Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing DFTPP (refer to table 12.2). The tune verification standard can be combined with the CCV standard provided that the amount of DFTPP introduced into the system meets the method criteria. For semi-volatile analysis, the system must also be verified for inertness **unless PAHs are the only class of analytes to be analyzed**. This is done simultaneously by the inclusion of DDT, benzidine and pentachlorophenol. DDT is used to verify breakdown conditions; benzidine and pentachlorophenol are used to check for tailing due to system activity.

12.2.1. Mass Axis Alignment / Ion Ration Verification

After the analysis of this standard, the mass spectrum of DFTPP must be evaluated against the following criteria:

Table 12.2: Tune Evaluation Criteria

Mass (m/z)	Mass Axis criteria	Ion Abundance criteria
51	Present	10.0-80.0% of m/z 198
68		<2.0% of m/z 69
69	Present	Present
70		<2.0% of m/z 69
127	Present	10.0-80.0% of m/z 198
197		<2.0% of m/z 198
198	Present	Base peak, >50% of Mass 442
199	Present	5.0-9.0% of m/z 198
275	Present	10.0-60.0% of m/z 198
365		>1% of m/z 198
441		Present, but less than m/z 443
442	Present	>50% of m/z 198
443	Present	15.0-24.0% of m/z 442

12.2.2. To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for DFTPP. If the software has a

program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one the following manners, in the listed order:

- 12.2.2.1 Using an average of three scans, centered on the apex of the peak; or,
- 12.2.2.2 Using an average of all scans across the width of the peak, taken at half height; or,
- 12.2.2.3 Using an average of all scans taken across the width of the peak from baseline to baseline.

A background scan taken immediately before but not including the peak must be subtracted.

12.2.3. Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the DFTPP tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the DFTPP tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

12.2.4. If the ratios do not meet the criteria, refer to the following corrective actions to address the problem:

12.3. Tailing Factor Verification-

12.3.1. Benzidine and Pentachlorophenol should be present at their normal responses, and peak tailing should not be to an excess.

12.4. **Breakdown Verification-** The GC/MS system must be sufficiently inert such that DDT will not breakdown excessively while in the injection port. The inertness is assessed by calculating the percent breakdown of DDT into the products DDD and DDE. The calculation is performed as follows:

$$\% \text{ DDT Breakdown} = \frac{(\text{Sum of responses for DDD and DDE})}{(\text{Sum of responses for DDT, DDD and DDE})} * 100$$

12.4.1. The % breakdown must not exceed 20%. If the breakdown of DDT exceeds this amount, samples may be analyzed if all requested analytes are within +/- 20% in the CCV (documented on an out-of-control form), otherwise corrective action must be taken prior to analysis of samples. The breakdown must be verified by the analysis of another breakdown standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

12.4.1.1 If the tune standard does not meet the criteria, refer to the following corrective actions to address the problem.

12.4.1.1.1 Retune the mass spectrometer following the equipment manufacturers' instructions. The tune status must be verified after the tuning procedures.

12.4.1.1.2 If this fails, change filament and retune.

12.4.1.1.3 If this fails, take down the mass spectrometer and clean the instrument.

12.5. Calibration Verification: After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 11.2.1 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

12.6. If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 11.4.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

12.7. Note: In situations where the instrument will run unattended (i.e. overnight), the analyst may load sequential CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, the CCV must be evaluated according to the protocol set forth in the Quality Assurance Manual within the Equipment and Measurement Traceability section.

12.8. Sample Preparation- Water Samples: Aqueous samples are prepared according to EPA 3510C. These procedures are contained in a separate standard operating procedure. Refer to SOP number S-GB-O-053, *Separatory Funnel Extraction by 3510C* (most current revision or replacement) for details on the preparation of aqueous samples.

12.8.1. Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution.

12.9. Sample Preparation- Soil Samples: Aqueous samples are prepared according to EPA 3546. These procedures are contained in a separate standard operating procedure. Refer to SOP number S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices by 3546* (most current revision or replacement) for details on the preparation of soil or solid samples.

12.9.1. Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution.

12.10. Dilutions

12.10.1. Dilutions on sample extracts must be prepared in a volumetric fashion. Sample aliquots should be taken in volumetric syringes and brought to volume by the addition of solvent via an appropriate syringe. In the event a dilution is made to bring a target analyte into calibration range, the analyst should make a dilution such that the target analyte is roughly the equivalent of the mid calibration point whenever possible. If dilutions are made on extracts that already contain internal standards, a proportional aliquot of internal standard solution must be added to the diluted extract based on the volume of diluent used.

12.11. Sample Analysis-

12.11.1. GC/MS System Preparation

12.11.1.1 Operating Parameters—Set up the instrument parameters shown in Table 12.1.

12.11.1.2 System Tuning and GC Performance Checks—Analyze the Tuning Solution and tune the mass spectrometer to meet the criteria shown in Section 12.2.2. Verify acceptable GC system performance is described in Section 12.2.2. Print out a tune report.

12.11.2. Batch Sequence—Generate a sequence to run a batch of samples.

12.11.2.1 Initial Calibration – The typical batch for initial calibration should include:

Tune Standard
Calibration Level 1
Calibration Level 2
Calibration Level 3
Calibration Level 4
Calibration Level 5
Calibration and System Performance Solution

12.11.2.2 Sample Analysis – The typical batch for sample analysis should include the following. Preparation LCS, MS, MSD, and Duplicate sample extracts is described in the appropriate sample preparation SOP.

Tune Standard
Calibration and System Performance Solution
Instrument Blank
Method Blank
Laboratory Control Sample
Laboratory Control Sample Duplicate
20 samples
Matrix Spike
Matrix Spike Duplicate

12.11.3. Autosampler – Load the autosampler with standards and samples for the batch created above.

12.11.4. Analyze Samples – Analyze all standards, quality control samples, and environmental samples.

12.12. Qualitative Analysis

12.12.1. **Retention Time Comparison:** The relative retention time (RRT) of the sample component must be within ± 0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co eluting components.

12.12.2. **Mass Spectrum Comparison:** The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

12.12.2.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

12.12.2.2. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.12.2.3. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.

12.12.2.4. Under SIM conditions, only those ions collected will be present in the spectrum. Therefore, the best benchmark for spectral comparison should be the spectra obtained from the opening CCV. Ion intensity ratios should agree within 30% of those obtained in the standard.

12.12.3. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

Table 12.3: Ions

Compound	CAS Number	Primary Ion	Secondary Ion
Naphthalene	91-20-3	128	129
2-Methylnaphthalene	91-57-6	142	141
1-Methylnaphthalene	90-12-0	142	141
Acenaphthylene	208-96-8	152	153
Acenaphthene	83-32-9	154	152
Fluorene	96-73-7	166	165
Phenanthrene	85-01-8	178	179
Anthracene	120-12-7	178	176
Fluoranthene	206-44-0	202	101
Pyrene	129-00-0	202	200
Benzo(a)anthracene	56-55-3	228	229
Chrysene	218-01-9	228	226
Benzo(b)fluoranthene	205-99-2	252	253
Benzo(k)fluoranthene	207-08-9	252	253
Benzo(a)pyrene	50-32-8	252	253
Indeno(1,2,3-cd)pyrene	193-39-5	276	138
Dibenzo(a,h)anthracene	53-70-3	278	139
Benzo(g,h,i)perylene	191-24-2	276	138
Benzo(e)pyrene	192-97-2	252	253
2-Fluorobiphenyl-SS	321-60-8	172	171
Terphenyl-d(14)-SS	98904-43-9	244	122
Naphthalene-d(8)-IS	1520-96-3	136	68
Acenaphthene-d(10)-IS	15067-26-2	164	162
Phenanthrene-d(10)-IS	1517-22-2	188	94
Chrysene-d(12)-IS	1719-03-5	240	120
Perylene-d(12)-IS	1520-96-3	264	260

12.13. Quantitative Analysis- Quantitation is based on the integrated abundance of the target analytes' quantitation ion using the internal standard technique.

Table 12.4: Internal Standard Assignments

Compound	Internal Standard
Naphthalene	Naphthalene-d(8)-IS
2-Methylnaphthalene	Naphthalene-d(8)-IS
1-Methylnaphthalene	Naphthalene-d(8)-IS
Acenaphthylene	Acenaphthene-d(10)-IS
Acenaphthene	Acenaphthene-d(10)-IS
Fluorene	Acenaphthene-d(10)-IS
Phenanthrene	Phenanthrene-d(10)-IS
Anthracene	Phenanthrene-d(10)-IS
Fluoranthene	Phenanthrene-d(10)-IS
Pyrene	Chrysene-d(12)-IS
Benzo(a)anthracene	Chrysene-d(12)-IS
Chrysene	Chrysene-d(12)-IS
Benzo(e)pyrene	Perylene-d(12)-IS
Benzo(b)fluoranthene	Perylene-d(12)-IS
Benzo(k)fluoranthene	Perylene-d(12)-IS
Benzo(a)pyrene	Perylene-d(12)-IS
Indeno(1,2,3-cd)pyrene	Perylene-d(12)-IS
Dibenzo(a,h)anthracene	Perylene-d(12)-IS
Benzo(g,h,i)perylene	Perylene-d(12)-IS
2-Fluorobiphenyl-SS	Acenaphthene-d(10)-IS
Terphenyl-d(14)-SS	Chrysene-d(12)-IS

13. Quality Control

13.1. Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples. If analyzing a TCLP sample, the associated TCLP Blank must also be analyzed with the batch.	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	Re-analyze blank to confirm failure. Qualify results and/or re-extract associated samples. <u>Exceptions:</u> If sample ND, report sample without qualification; If sample result >10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination; If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Full Target List compounds	One per batch of up to 20 samples. LCSD is performed for water samples where no volume is provided for MS/MSD. In this instance, the batch data must be qualified with an M5 data qualifier.	Laboratory derived limits <u>Full Target List:</u> Marginal exceedances allowed according to the TNI standard.	Re-analyze the LCS to verify failure; If LCS passes, review samples for potential injection problems; If problem persists, check spike solution; Re-extract samples where possible. <u>Exceptions:</u> If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Full Target List compounds	One per batch of up to 20 samples. If analyzing TCLP samples, one MS must be analyzed for each TCLP matrix.	Laboratory derived limits	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences.
MSD / Duplicate	MS Duplicate <u>OR (alternative)</u> Sample Dup	One for every 5% of all environmental samples	Laboratory derived Limits	Report results with an appropriate footnote.

13.2. Table 13.2 – Sample Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	Retention Time: RT must be ± 30 seconds from last calibration check on all samples	Retention Time Failure: If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting; Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	2-Fluorobiphenyl Terphenyl-d14	Added to all samples, spikes, control samples and method blanks prior to analysis	Laboratory derived limits	Re-analyze extract to confirm failure. Assess impact of sample matrix. In the absence of obvious matrix interference (high background, extremely dark extract), re-extract sample. <u>Exceptions:</u> Surrogate recovery above criteria and target compounds < RL, result may be reported with appropriate footnote. Surrogate recovery out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote.

14. Data Analysis and Calculations

14.1. **Raw Data Results:** The GC/MS data system will calculate the concentration of each analyte in the sample extract. If supplied with the preparation parameters, the system may be able to calculate the results back to the original matrix. The calculation for the concentration of the target analyte in the original matrix is listed below and is based on the calibration table in units of ppm (ug/mL). If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

14.1.1. Calculate response factors (RFs) for each compound as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

Where: A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specified internal standard.

C_{is} = Concentration of the specified internal standard (ug/mL).

C_x = Concentration of the compound being measure (ug/mL).

14.1.2. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{s}{\bar{X}} \times 100\%$$

Where: s = Standard Deviation of initial response (see Section 14.1.1)
X = Mean of the Response Factors in Section 14.1.1

14.1.3. The Standard Deviation is calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}}$$

Where: x_i = Each individual response factor
N = Number of the Response factors mentioned above.

14.1.4. The Relative Percent Difference (%D) is calculated as follows:

$$\%Difference = \frac{|RF_i - RF_c|}{RF_i} \times 100$$

Where: RF_i = Average response factor from initial calibration
 RF_c = Response factor from current verification check standard.

14.1.5. Results Calculation- Aqueous Samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(C_x)(V_x)(DF)}{(V_s)}$$

Where: C_x = Concentration in extract ($\mu\text{g/mL}$).
 V_v = Volume of final extract (mL).
DF = Dilution factor.
 V_s = Volume of water sample extracted (mL).

14.1.6. Results Calculation- Soil/Solid Samples:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(C_x)(V_x)(1000)(DF)}{(W_s)}$$

Where: C_x = Concentration in extract ($\mu\text{g/mL}$).
 V_v = Volume of final extract (mL).
DF = Dilution factor.
 W_s = Weight of soil sample extracted (g).

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Tables 12.2, 13.1 and 13.2.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Tables 12.2, 13.1 and 13.2.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Tables 12.2, 13.1 and 13.2.

18. Method Performance

18.1. **Method Detection Limit (MDL) Study:** An MDL study must be conducted annually per S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement) for each matrix per instrument.

18.2. **Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement).

19. Method Modifications

19.1. Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

19.2. All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

19.3. Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into the document as minimum requirements for Pace laboratories.

19.4. The Low Volume Extraction, High Volume Injection is a modification which the sample volume used for extraction purposes is reduced to 100 mL , and the injection volume into the analytical column is 50uL. The injection volume is the same for all standards and sample extracts.

19.5. The laboratory follows the DFTPP Tune criteria outlined in EPA 525.2.

19.6. If a client fails to provide the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument operations manual or the SOP S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance* (current revision or replacement).

21. Troubleshooting

21.1. Please refer to the instrument manufacturer operations manual.

22. Safety

22.1. **Standards and Reagents:** The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets

(SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples:** Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. **Equipment:** Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The GC pneumatic system uses gas under high pressure. This high pressure introduces the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. USEPA, SW-846, Method 8270C, “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

25.2. USEPA, SW-846, Method 8000B, “Determinative Chromatographic Separations”, December 1996.

25.3. USEPA, Method 525.2, Revision 2.0 (1995), “Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry”.

25.4. Pace Quality Assurance Manual- most current version.

25.5. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, “Quality Systems”- most current version.

25.6. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-GB-O-050-Rev.01	Updated SOP reference	07Jul2011
SOT-ALL-O-008-rev.01	<p>Converted to current SOT format.</p> <p>Section 6: removed definitions for run sequence and tune period.</p> <p>Section 6.1: added sections in red letter text for local edits.</p> <p>Tables 7.1: changed to red text to allow for local lab edits.</p> <p>Table 9.1: changed to Reagents and Stock Standards so that stock standard catalog information could be captured.</p> <p>Table 9.2: changed some sections to red text to allow for local lab edits.</p> <p>Table 10.1: changed compound names to red letter text to allow for local lab edits.</p> <p>Section 10.4.3: changed list of corrective actions to red text to allow for local lab edits.</p> <p>Section 11.13.1: changed "middle" to "upper half".</p> <p>Sections 11.14 and 11.15: changed calculations to match 8270 SOT (apparently the last revision had the 8260 calculations).</p> <p>Section 13.3: removed explanation of method modifications section.</p> <p>Section 15: added references 15.3-15.5.</p>	03Aug2011
S-GB-O-050-Rev.02	<p>Throughout document: Incorporated information regarding the analysis of High Volume Injections for PAH analysis.</p> <p>Table 12.1: Included information specific to TCLP analysis requirements.</p> <p>Table 12.2: Removed erroneous internal standard: 1,4-Dichlorobenzene-d4. Removed erroneous surrogates: Nitrobenzene-d5, Phenol-d6, 2-Fluorphenol and 2,4,6-Tribromophenol.</p> <p>Section 11.13.1: Added calculations.</p> <p>Section 13.3: Added Method Modification section, included information for modification to HVI Method.</p>	03May2012
S-GB-O-050-Rev.03	<p>Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 <i>Preparation of SOPs</i>.</p> <p>Throughout Document: Updated table references to coincide with their proper number format.</p> <p>Table 11.2: Updated DFTPP Tune Criteria to be consistent with EPA 525.2.</p> <p>Section 11.3.2 and Table 11.4: Updated ICV criteria to match CCV criteria and calculations.</p> <p>Section 25: Added EPA 525.2 reference.</p>	27Jan2014

ATTACHMENT B3

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

Title: Multi-Site QAPP**Revision Number: 2****Addendum Date: 7/23/14****Page 1 of 3****Reference Limits and Evaluation Table**

Laboratory: Pace

Matrix: Water

Analytical Group/Method: Semivolatile Organic Compounds - 8270C SIM High Volume Injection

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit ² (applicable units)	Achievable Laboratory Limits ¹	
				MDLs	QLs
4-Nitroaniline	100-01-6	3.4			
4-Nitrophenol	100-02-7	no value			
Benzaldehyde	100-52-7	3700			
4-Bromophenylphenyl ether	101-55-3	no value			
Caprolactam	105-60-2	18000			
2,4-Dimethylphenol	105-67-9	100			
3&4-Methylphenol (m&p)	106-44-5	67			
4-Methylphenol	106-44-58	25			
4-Chloroaniline	106-47-8	28			
2,2'-Oxybis(1-chloropropane)	108-60-1	0.32			
Phenol	108-95-2	100			
bis(2-Chloroethyl) ether	111-44-4	10			
bis(2-Chloroethoxy)methane	111-91-1	110			
bis(2-Ethylhexyl)phthalate	117-81-7	6			
Di-n-octylphthalate	117-84-0	140			
Hexachlorobenzene	118-74-1	0.06			
Anthracene	120-12-7	0.059			
2,4-Dichlorophenol	120-83-2	21			
2,4-Dinitrotoluene	121-14-2	0.02			

Pyrene	129-00-0	0.29			
Dimethylphthalate	131-11-3	no value			
Dibenzofuran	132-64-9	37			
Atrazine	1912-24-9	3			
Benzo(g,h,i)perylene	191-24-2	0.013			
Indeno(1,2,3-cd)pyrene	193-39-5	0.008			
Benzo(b)fluoranthene	205-99-2	0.019			
Fluoranthene	206-44-0	0.2			
Benzo(k)fluoranthene	207-08-9	0.018			
Acenaphthylene	208-96-8	8.77			
Chrysene	218-01-9	0.058			
Benzo(a)pyrene	50-32-8	0.027			
2,4-Dinitrophenol	51-28-5	14			
4,6-Dinitro-2-methylphenol	534-52-1	2.9			
Dibenz(a,h)anthracene	53-70-3	0.008			
Benzo(a)anthracene	56-55-3	0.064			
2,3,4,6-Tetrachlorophenol	58-90-2	1100			
4-Chloro-3-methylphenol	59-50-7	3700			
2,6-Dinitrotoluene	606-20-2	0.31			
N-Nitroso-di-n-propylamine	621-64-7	1.8			
Hexachloroethane	67-72-1	7			
4-Chlorophenylphenyl ether	7005-72-3	no value			
Hexachlorocyclopentadiene	77-47-4	50			
Isophorone	78-59-1	1400			
Acenaphthene	83-32-9	1.6			
Diethylphthalate	84-66-2	5600			
Di-n-butylphthalate	84-74-2	700			
Phenanthrene	85-01-8	0.55			
Butylbenzylphthalate	85-68-7	1400			
N-Nitrosodiphenylamine	86-30-6	3.2			
Fluorene	86-73-7	1.12			
Carbazole	86-74-8	no value			
Hexachloro-1,3-butadiene	87-68-3	0.86			
Pentachlorophenol	87-86-5	1			
2,4,6-Trichlorophenol	88-06-2	10			
2-Nitroaniline	88-74-4	370			
2-Nitrophenol	88-75-5	no value			
Naphthalene (cancer)	91-20-3	0.6			

2-Methylnaphthalene	91-57-6	150			
2-Chloronaphthalene	91-58-7	2900			
3,3'-Dichlorobenzidine	91-94-1	20			
Biphenyl (Diphenyl)	92-52-4	1800			
2-Methylphenol (o-Cresol)	95-48-7	350			
2-Chlorophenol	95-57-8	35			
1,2,4,5-Tetrachlorobenzene	95-94-3	11			
2,4,5-Trichlorophenol	95-95-4	700			
Acetophenone	98-86-2	3700			
Nitrobenzene	98-95-3	3.5			
3-Nitroaniline	99-09-2	2.1			
Perylene	NA	0.026			

¹Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. Limits vary over time per lab performance tests and capabilities. As Multi-Site Screening Levels/Project Quantitation Limits are updated, lab's ability to achieve new limits are verified before work proceeds.

²Project Quantitation Limits/Multi-Site Screening Levels are updated approximately every six months per the USEPA-approved Multi-Site Risk Assessment Framework for the Integrys Multi-Site Manufactured Gas Plant Program. As Multi-Site Screening Levels/Project Quantitation Limits are updated, lab's ability to achieve new limits are verified before work proceeds.

ATTACHMENT B4

**QAPP Worksheet #24
(UFP-QAPP Manual Section 3.2.2)**

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference¹
GC/MS	Tune (BFB, DFTPP)	Prior to initial calibration or continuing calibration; every 12 hours	Refer to SOP	Correct problem; re-analyze tune	Analyst	
GC/MS	Initial Calibration	Prior to sample analysis or as needed	VOC : CCC \leq 30% RSD; SPCC \geq 0.300 or 0.100; all other targets <15% RSD; linear $r \geq$ 0.995 SVOC: CCC \leq 30% RSD; SPCC \geq 0.05; all other targets grand mean <15% RSD; linear $r \geq$ 0.995	Correct problem; repeat initial calibration	Analyst	
GC/MS	Continuing Calibration	Daily, before sample analysis and every 12 hours of tune time	VOC : CCC \leq 20% DIFF/Drift SPCC \geq 0.300 or 0.100; SVOC: CCC \leq 20% DIFF/Drift SPCC \geq 0.05	Correct problem and repeat CCV and associated samples; repeat initial calibration if necessary and CCV and samples; may report non-detects if biased high.	Analyst	
Metals (ICP)	Initial Calibration	Daily initial calibration prior to sample analysis	$r \geq$ 0.995	Correct problem; repeat initial calibration	Analyst	
Metals (ICP)	Continuing Calibration	After every 10 readings and end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements and bracketed samples; may report non-detects if biased high	Analyst	

¹ Lab Notes: See Quality System Manual and specific Analytical Standard Operating Procedures (SOPs) for full details

**QAPP Worksheet #25
(UFP-QAPP Manual Section 3.2.3)**

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference¹
Hewlett Packard GC/MS	Routine	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	As required	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	
Thermo Scientific ICP	Routine	Inspect sample introduction system, nebulizer, torch and injection tubing	Check for salt build up, dirt and debris that may restrict flow	As required	Passing calibration	Perform maintenance, replace/clean tubing, check standards, recalibrate	Laboratory Analyst	

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

Lab Note: All maintenance procedures will follow the Laboratory Quality System Manual and specific Analytical Standard Operating Procedures (SOP)

ATTACHMENT C1

SOP Number/Revision No.: **3510 / NV03-24.13** Effective Date: 3/29/2013

Last Mod. Date: 12/31/2012

SOP Title: **Method 3510C, Separatory Funnel Liquid-Liquid Extraction**

Affected SOP Section Number(s): Section 10.3, Sample Extraction (non-fluff), and Section 10.4, Sample Concentration by Kuderna-Danish Technique.

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 03P

Revision Number with Mod ID: **13a**

*The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

- ☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.
- ☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.
- ☒ Procedural Changes (Define Below) – Re-Training Required.
- ☐ Other

2. Summary of Procedure Change: **Delete crossed out, add underlined.**

Section 10.3, Sample Extraction (non-fluff), Step 7

Dry the extract by passing it through a Teflon™ funnel containing about 2/3 full of pre-rinsed, anhydrous Sodium sulfate. Collect the dried extract in a clean Erlenmeyer flask. Rinse the funnel ~~Erlenmeyer flask~~, which contained the solvent extract, with 20-30 mL Methylene chloride and add it to the flask ~~funnel~~ to complete the quantitative transfer. Perform the concentration using the Kuderna-Danish technique.

Section 10.4, Sample Concentration by Kuderna-Danish technique, Steps 2, 6, and 8

- | | |
|---|--|
| 2 | Add one or two clean boiling chips to the <u>KD flask</u> . Quantitatively transfer extract to the <u>KD</u> . <u>Rinse the flask with approximately 10-20 mL solvent.</u> and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of Methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15 – 20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the temperature and/or flask position to complete the concentration in 10 - 20 minutes. Record the temperature. At the proper rate of distillation, the balls of the column actively chatter, but the chambers do not flood. When the apparent volume of liquid reaches about 10 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. |
|---|--|

Nitrogen blow-down technique	
6	Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to the just below final volume indicated in Table 1, using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). Bring the extract to the required final volume using Class A volumetric flasks.
8	The sample is then transferred to a Class A volumetric flask and adjusted to the final volume <u>by</u> using the rinsate from the tube using the last solvent used.




<i>Gaerby Reiser</i>	3/21/13	<i>Joly D. J.</i>	3/21/13
Department Manager Approval	Date	Operations Manager Approval	Date
<i>Michael H. Dunn</i>	3/21/13		
Technical Approval	Date		
Quality Manager Approval			

SOP Number/Revision No.: **3510 / NV03-24.13**

Effective Date: 3/29/2013

Revision Number with Mod ID: **13a**

**Title: SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION
SW-846 METHOD 3510C**

Approvals (Signature/Date)	
	12/17/12
Jacoby Robinson Department Manager	Date
	11/27/12
Johnny Davis Extractions Operations Manager Health & Safety Manager / Coordinator	Date
	11/20/12
Michael H. Dunn Technical Director Quality Assurance Manager	Date

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1.0 Scope and Application

1.1 Analyte, Matrices: This method predominantly describes a procedure for extracting water-insoluble and slightly water-soluble organic compounds from aqueous samples and concentrating them for injection onto a gas chromatograph set up for an appropriate determinative method.

1.2 Reporting Limits: Results are dependent on the volume used, degree of contamination, ability to concentrate, and the sensitivity of the determinative method.

1.3 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Manager. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

A measured volume of sample, nominally 1000, 250, or 125 mL, at a specified pH, is serially extracted with Methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used.

3.0 Definitions

See Appendix 5 of TestAmerica Nashville's QA Manual for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

Interferences are often due to contamination from solvents or extraction glassware.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements: Use the hoods to evacuate solvent vapors from the building and dispose of solvent wastes appropriately.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methylene chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m ³	This material causes burns in contact with the skin or eyes. Inhalation of Sodium Hydroxide dust causes irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrator	1 mg/m ³	This material causes burns if comes into contact with the skin or eyes. Inhalation of vapors causes irritation of the nasal and respiratory system.
Acetonitrile	Flammable Poison	40 ppm TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Kuderna-Danish (K-D) apparatus.
 - Concentrator tube, 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts. \
 - Evaporation flask, 250 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.
 - Snyder column, Three-ball macro (Kontes K-503000-0121 or equivalent).
 - Snyder column, 3 chamber micro (Kontes K-569001-0219 or equivalent).
 - Clamps
- Nitrogen Evaporator: N-Evap Model #116 by Organomation, or equivalent.

6.2 Supplies

- Separatory funnel, 2 liter, 500 mL, or 250 mL, Teflon™ with polytetrafluoroethylene (PTFE) stopcock.
- Funnel, Teflon™. Put a plug of glass wool in a funnel and fill about 2/3 full with sodium sulfate. Rinse funnel and Sodium sulfate with 5-10 mL of Methylene chloride before use.
- Boiling chips, solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).
- Water bath, heated, capable of temperature control ($\pm 5^{\circ}\text{C}$). The bath is used in a hood.
- Vials, glass with PTFE-lined screw-caps.
- pH indicator paper, 0 – 14 pH range.
- Erlenmeyer flask, Teflon™ 250 mL or 500 mL.
- Graduated cylinder, glass, Class A, 1 liter, or equivalent.
- Volumetric flasks, Class A, at 1, 5, and 10 mL.
- Centrifuge, capable of approximately 2000 rpm.
- Nitrogen, compressed gas, high purity.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents must conform to the specifications of the Committee on Analytical Reagents of the America Chemical Society, where such specifications are available. Other grades may be used, however, provided it is first ascertained that the reagent is of sufficiently high purity to

permit its use without lessening the accuracy of the determination. Reagents are stored in glass or Teflon™ to prevent the leaching of contaminants from plastic containers.

7.2 Reagent water, analyte-free.

7.3 Sodium hydroxide solution (10 N), NaOH, commercial source.

7.4 Sodium sulfate (granular, anhydrous), Na₂SO₄. Purify by heating to about 400°C for about 4 hours in a shallow tray. Store in a sealed, glass container. A commercially baked product is acceptable.

7.5 Sulfuric acid solution, H₂SO₄, commercial source. Make a 1:1 dilution with reagent water.

7.6 Extraction/exchange solvents: All solvents are pesticide quality or equivalent and from commercial sources: **Methylene chloride**, CH₂Cl₂, **Hexane**, C₆H₁₄, **Acetonitrile**, CH₃CN.

7.7 Sodium chloride (NaCl), commercial source, granular.

7.8 Acetone, CH₃COCH₃, commercial source.

7.9 Spiking Solutions: See the determinative method and LIMS for information. These are purchased ready for use or prepared in the analysis department.

7.10 See SOP Reagent and Standard Purchase, Preparation, control, Documentation / NV08-214 for information on shelf-life and storage requirements for standards and reagents.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time from Collection	Reference
Water	Amber Glass, 1 L, 250 mL, or 125 mL Teflon™-lined cap	varies	0-6°C	7 days until extraction, 40 days until analysis	SW846 Chapter 2

9.0 Quality Control

Refer to the quality control section of TestAmerica Nashville's QA Manual for specific quality control (QC) policies. The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

The following quality control samples are prepared with each batch of no more than 20 samples.	
Quality Controls	Frequency
Method Blank	1 in 20 or fewer samples
Laboratory Control Sample (LCS) ¹ , second source	1 in 20 or fewer samples
Matrix Spike	1 in 20 or fewer samples
Matrix Spike Duplicate	1 in 20 or fewer samples

¹For AZ, TX, WV samples, a LCS duplicate is required.

- **Method blank:** The laboratory prepares and analyzes a method blank (reagent water) with each batch.
- **A Laboratory Control Sample (LCS),** reagent water spiked with a source different from the calibration standard, is analyzed with every batch. Use the same sample preparations, analytical methods, and QA/QC procedures employed for the test samples.
- **Matrix Spike/Matrix Spike Duplicate:** Sample homogeneity and the chemical nature of the sample matrix can affect analyte recovery and the quality of the data. Taking separate aliquots from the sample for replicate and fortified analyses can in some cases assess the

effect. Unless otherwise specified by the data user, the MS/MSD procedure is required.

- The laboratory must add a known amount of each analyte to be reported to a minimum of 5% of the routine samples. In each case the MS/MSD aliquot must be a duplicate of the aliquot used for sample analysis and added **prior** to sample extraction.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	1000, 250 , or 125 mL

1	Generally, the entire contents of the sample bottle are to be extracted. Mark the level of sample on the outside of the bottle and measure against a calibrated bottle of the same size and shape. (Bottles are calibrated quarterly using Class A volumetric flasks. See Section 17.3.) Alternatively, using a Class A, graduated cylinder, measure the required amount of sample. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1 L with organic-free reagent water, or samples may be collected in smaller sample bottles and the whole sample used. Shake the sample container well and transfer to a separatory funnel that has been pre-rinsed with about 10 mL of Methylene chloride.
2	For TCLP: 500 mL of the TCLP extract is used for semivolatile (BNA) extraction, 100 mL is used for pesticide extraction. For herbicide extraction, use 5 mL of TCLP extract. Reagent water is used to bring the volume to about 1 L. Add surrogates (Table 2) to all samples and QC. <ul style="list-style-type: none"> • For TCLP BNA, spike the LCS, MS/MSD with 1 mL of the TCLP BNA spike. • For TCLP Pesticides, spike with 0.5 mL TCLP Pesticides spike and surrogate and 1 mL of Toxaphene. • For TCLP herbicides, spike with 1.0 mL of TCLP Herbicide spike.
3	Mixing by shaking is sometimes ineffective as solids settle during the time required to secure the sub-sample aliquot for analysis. Usually decantation prior to shaking or after initial settling following mixing (in order to preserve the suspended solids) is not an appropriate homogenization procedure. However, if decantation is used, documentation of the process must be made in the preparation or analysis logbook or benchsheet. If the inclusion of solid material adversely affects the extraction or analysis procedure, notify the project manager. The project manager must contact the client to verify if they would like the lab to extract only the aqueous portion. In this case, pour out only the liquid portion into a graduated cylinder and note the volume decanted and the approximate percentage of sediment present on the benchsheet.
4	Using a glass syringe, add the surrogate spiking solution into each sample in the separatory funnel and mix well. (See Table 2 for details on the surrogate standard solution. This addition of surrogate must be made into both client and QC samples.)
5	Using a glass syringe, add the matrix spike/LCS spiking solution into the appropriately designated separatory funnels and mix well. (See Table 2, the determinative method, and LIMS for details on the matrix spike/LCS solution.)
6	Check the pH of the sample by immersing a glass or Teflon™ rod tip or a pipet in the sample and touch to wide-range pH paper and adjust the pH, if necessary, to the pH indicated in Table 1 , using 1:1 (v/v) Sulfuric acid or 10 N Sodium hydroxide. Rinse the glass or Teflon™ rod with Methylene chloride into the separatory funnel. Other strengths of acid or base solution may be employed, provided that they do not result in a significant change (< 1%) in

	the volume of sample extracted.
7	For Method 8270 BNA, always adjust to acid pH first. If only PAHs are being analyzed by Method 8270, then only the base-neutral extraction needs to be done. In order to extract only the base-neutral, all samples within the batch must be PAH only, and the QC must be treated the same as the samples.
8	QC samples are prepared using 1 liter organic-free reagent water.

10.2 Sample Extraction

1	Use Methylene chloride to rinse the graduated cylinder (or sample container) and transfer this rinse solvent to the separatory funnel containing the sample. Record the sample volume on the benchsheet. For 1 L sample volume, use 60 mL Methylene chloride. For 250 mL sample volume, use 15 mL Methylene chloride. For 125 mL sample volume use 8 mL Methylene chloride.
2	Seal and shake the separatory funnel vigorously for 1 - 2 minutes with periodic venting to release excess pressure. Methylene chloride creates excessive pressure very rapidly; therefore, initially vent immediately after the separatory funnel has been sealed and shaken once. Vent the separatory funnel into a hood to avoid exposure of the analyst to solvent vapors.
3	Allow the organic layer to clearly separate from the water phase. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation at approximately 2000 rpm for about 5 minutes, or other physical methods. Collect the solvent extract in an Erlenmeyer flask.
4	Repeat the extraction two more times using fresh portions of solvent. Combine the three solvent extracts.
5	If further pH adjustment and extraction is required, adjust the pH of the aqueous phase to the desired pH indicated in Table 1 (Section 17) . Serially extract three times with proportional volumes of Methylene chloride. Collect and combine the extracts and label the combined extract appropriately.
6	If performing GC/MS analysis (Method 8270), the acid/neutral and base extracts are combined prior to concentration. However, in some situations, separate concentration and analysis of the acid/neutral and base extracts may be preferable (e. g., if for regulatory purposes the presence or absence of specific acid/neutral or base compounds at low concentrations must be determined, separate extract analyses may be warranted).
7	Dry the extract by passing it through a Teflon™ funnel containing about 2/3 full of pre-rinsed, anhydrous sodium sulfate. Collect the dried extract in a clean Erlenmeyer flask. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of Methylene chloride and add it to the funnel to complete the quantitative transfer. Perform the concentration using the Kuderna-Danish Technique.
8	Extracts may be held prior to concentration if stored covered; in addition, 8310, 8270, and 8081 extracts when held are stored at 0-6°C and in the dark. Proceed to sample concentration.

10.3 Sample Concentration by Kuderna-Danish Technique

1	Assemble a K-D concentrator by attaching a 10-mL concentrator tube to a 250-mL, or smaller, evaporation flask.
---	--

2	Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of Methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (15 – 20°C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the temperature and/or flask position to complete the concentration in 10 - 20 minutes. Record the temperature. At the proper rate of distillation, the balls of the column actively chatter, but the chambers do not flood. When the apparent volume of liquid reaches about 10 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.
3	If a solvent exchange is required (as indicated in Table 1), remove the Snyder column and KD, and using nitrogen blow-down technique, reduce the volume to about 2 mL. To 4 mL of Hexane or 2 mL of Acetonitrile, whichever is the appropriate exchange solvent, add a new boiling chip, and attach the 3-chamber micro-Snyder column to the concentrator tube. Concentrate the extract and alter the temperature of the water bath, if necessary, to maintain proper distillation.
4	Remove the Snyder column and rinse it and its lower joints into the concentrator tube with 1 - 2 mL of Methylene chloride or exchange solvent. The extract is further concentrated by using the technique outlined in Section 10.3.6 or adjusted in a Class A volumetric to 1.0 - 10.0 mL with the solvent last used.
5	If further concentration is indicated in Table 1, use the nitrogen blow-down technique to adjust the extract to the final volume required.
Nitrogen blow-down technique	
6	Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to the just below final volume indicated in Table 1, using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). Bring the extract to the required final volume using Class A volumetric flasks.
7	The internal wall of the tube must be rinsed several times with Methylene chloride or appropriate solvent during the operation. During evaporation, the tube must be positioned to avoid water condensation (i. e., the solvent level is below the level of the water bath). Under normal procedures, the extract must not be allowed to become dry.
8	The sample is then transferred to a Class A volumetric flask and adjusted to the final volume using the last solvent used.
9	Transfer the sample to a vial with a PTFE-lined cap and label appropriately. Individual states may require silica gel clean-up. Refer to Table 1, state-specific SOPs and/or 8015 / NV05-31 for details. The extract may now be analyzed for the target analyses using the appropriate determinative technique(s). Store refrigerated.

10.4 Example Analysis Queue / Sequence

See the determinative method.

11.0 Calculations / Data Reduction

Enter sample volume and final extract volume in LIMS.

12.0 Method Performance

12.1 Method Detection Limits (MDLs): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses

performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.1 Wastestreams produced by this method:

- Extracted aqueous samples are collected, neutralized to a pH between 2.0 and 10.0, and discharged to the sanitary sewer.
- Used sodium sulfate and glass wool or filter paper contaminated with Methylene chloride from the extract drying step are placed in a hood overnight, then discarded in the trash.
- Assorted flammable solvent waste from various rinses is collected in the flammable waste drums.

15.0 References / Cross-References

15.1 SW-846 Method 3510C, Update III, Revision 3, December 1996.

15.2 CA LUFT Manual, Version 2.0, October 4, 2010.

TestAmerica Nashville's Quality Assurance Manual.

15.3 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.4 SOPs: Waste Disposal / NV10-83, 8270 / NV04-22, 8081 / NV04-16, 8082 / NV04-105, 8015 / NV05-31, 8310 / NV04-57, FL PRO / NV04-78, WI DRO / NV04-38, MADEP-EPH / NV04-168, NWTPH-Dx / NV04-190, NWTPH-EPH / NV04-191, OA-2 / NV04-188, OK DRO / NV04-74, TN EPH / NV04-187, CT ETPH / NV04-86, Training Procedures for Environmental Technical Staff / NV08-199, Balance Calibration / NV08-213, Reagent and Standard Purchase, Preparation, control, Documentation / NV08-214.

15.5 Controlled Documents: PF-1, Prep Lab Summary Chart, QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

16.0 Method Modifications

Item	Modification
1	Use of reduced sample volumes.

17.0 Attachments

17.1

Table 1. Specific Extraction Conditions for Various Determinative Methods

Determinative Method	Initial Extraction pH	Secondary Extraction pH	Exchange Solvent Req. for Analysis	Exchange Solvent Required for Cleanup	Volume of Extract Required for Cleanup (mL)	Final Extract Volume for Analysis (mL) ^a
8081	5-9	None	Hexane	Hexane	5.0	5.0
8082	5-9	None	Hexane	Hexane	5.0	5.0
8270 ^a	<2	>11	None	-	-	1.0
8310	As rec'd	None	Acetonitrile	Acetonitrile	-	1.0
8015	None	None	None	-	-	1.0
CA TPH (LUFT)	<2	None	None			1.0 ^b
CT-ETPH	<2	None	None	None		1.0
FL PRO	<2	None	None	-	2.0	2.0 ^b
MA-EPH	<2	None	None	Hexane	1.0	1.0 ^b
NWTPH-Dx	<2	None	None		1.0	1.0 ^b
NWTPH-EPH	<2	None	None	Hexane	2.0	2.0 ^b
OA-2	<2	None	None	-	-	1.0
TN-EPH	<2	None	None	-	-	1.0
WI/OK-DRO	<2	None	None	-	-	1.0

^a If only PAHs are being analyzed by Method 8270, then only the base extraction needs to be done. In order to do this, all samples within the batch must be PAH only and the QC **must** be treated the same as the samples.

^b Silica gel cleanup required.

17.2

Table 2. Surrogate and Matrix Spike/LCS Amounts

Method	Surrogate Spike Amount	MS/LCS Spike Amount	Sample Volume
8081	1.0 mL of Pest/PCB surrogate	1.0 mL Pest spike, 1.0 mL Toxaphene/Technical chlordane spike	1 L
8081	200 µL of Pest/PCB surrogate	200 µL Pest spike, 200 µL Toxaphene/Technical chlordane spike	125 mL
8082	1.0 mL of Pest/PCB surrogate	1.0 mL PCB spike	1 L
8082	200 µL of Pest/PCB surrogate	200 µL PCB spike	125 mL
8270	1.0 mL of BNA surrogate	500 µL of BNA spike	1 L
8270	200 µL of BNA surrogate	100 µL of BNA spike	250 mL

Method	Surrogate Spike Amount	MS/LCS Spike Amount	Sample Volume
8310	1.0 mL of HPLC surrogate	1.0 mL of HPLC spike	1
8015	1.0 mL of o-Terphenyl surrogate	1.0 mL DRO spike	1 L
8015	0.200 µL of o-Terphenyl surrogate	200 µL DRO spike	250 mL
CA TPH (LUFT)	1.0 mL of o-Terphenyl surrogate	1.0 mL DRO spike	1
CT ETPH	1.0 mL of o-Terphenyl surrogate	2.0 mL of FL/WI spike	1
FL PRO	2.0 mL of o-Terphenyl surrogate 2 mL of C ₃₅ surrogate	2.0 mL of FL/WI spike	1
MA-EPH	1 mL of MA surrogate 1 mL fractionation surrogate	1 mL of MA spike	1
NWTPH-Dx	1.0 mL of o-Terphenyl surrogate	1 mL of DRO spike	1
NWTPH-EPH	2 mL o-Terphenyl surrogate	2 mL MA spike	1
OA-2	1 mL o-Terphenyl surrogate	1 mL DRO spike	1
OK-DRO	1 mL of o-Terphenyl surrogate	2.0 mL of FL/WI spike	1
TN-EPH	1 mL o-Terphenyl surrogate	1 mL TN EPH spike	1
WI/OK-DRO	1.0 mL of C ₃₅ surrogate	2.0 mL of FL/WI spike	1
See individual SOPs for information on compounds, concentrations, and how to make the surrogate and spike solutions. Also, see the Prep Summary Chart for additional spike and surrogate information.			

17.3 Calibration of Bottles: Representative bottles, of varying size and shape, are calibrated using 100 mL, 50 mL, and 10 mL Class A volumetric flasks. Graduation marks are made accordingly, and bottles are then ready to be used as measuring guides for liquid samples. Date of calibration is noted on the bottle. The calibrated bottles are verified for accuracy quarterly and the verification recorded in a logbook.

18.0 Revision History

- Revision 8, dated 30 April 2008
 - Integration for TestAmerica and STL operations.
- Revision 9, dated 25 September 2009
 - Ohio VAP requirements
- Revision 10, dated 29 January 2010.
 - Addition of centrifuge to Section 6.2.
 - Define acronyms, abbreviations and/or refer to QAF-45.

- Add Section 14.2 to document.
- Change sample volume to 1000 mL.
- Revision 11, dated 30 June 2011.
 - Addition of Nitrogen to list of supplies (Amendment 10a).
 - Change sample volume for 8081/8082 to 1 Liter.
 - Clarify TCLP 8151 spike amounts.
 - Remove vertical adjustment of the apparatus in the water bath from sample concentration procedure description.
 - Distinguish Hexane and Acetonitrile volume additions when solvent exchange is required.
 - Organizational changes.
 - Change glass funnels to Teflon™ funnels.
- Revision 12, dated 30 April 2012
 - Organizational changes.
 - Addition of change form 11a, removing C35 surrogate from NWTPH-EPH.
 - Add considerations for samples with large amounts of solids, and revise sequence of steps for sample preparation.
 - Noted bottle calibration quarterly frequency in Section 10. Added date of calibration noted on bottle in section 17.3.
- Revision 13, dated 31 December 2012
 - Organizational changes.
 - Add information for Reduced Volume Extraction / Low Volume Injection.

ATTACHMENT C2

SOP Number/Revision No.: **8270 / NV/SA04-22.15**

Effective Date: **3/29/2013**

Last Mod. Date: 12/31/12

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Affected SOP Section Number(s): Section 3.0, Definitions; Section 7.0, Reagents and Standards, Section 9.1, Sample QC, Section 10.2, Calibration, Section 16.0, Method Modification

CONTROLLED DISTRIBUTION

ISSUED TO: QA Server, 04B

Revision Number with Mod ID: **15a**

*The following SOP change is in effect as of the stated date. This form will remain attached to the referenced SOP until such a time that the SOP is updated, approved, and redistributed, at which time it will become part of the historical SOP record. **Append this form to the front of the SOP copy.***

1. Reason for SOP Change:

- ☐ Typographical Corrections (Non-Technical) – Re-Training Not Required.
- ☐ Typographical Corrections (Technical – Define) – Analyst acknowledgement of corrections is required.
- ☒ Procedural Changes (Define Below) – Re-Training Required.
- ☐ Other

2. Summary of Procedure Change: Add bold text, delete crossed-out text.

Section 3.0, Definitions: Add a last sentence to 3.1 Reduced Volume Extraction / Large Volume Injection (RVE/LVI): **Generally, reduce all concentrations by a factor of RVE/LVI, i. e., 5.**

Section 7.0, Reagents and Standards

7.6 GC/MS Tuning Standard: Add to the first sentence: "A Methylene chloride solution containing 50 µg/mL **[RVE/LVI: 10 µg/L]** of Decafluorotriphenylphosphine (DFTPP) is prepared.

7.7 Surrogate Standards: To the bullet item, add a last sentence: **Dilute by five for RVE/LVI.**

Section 9.1, Sample QC, Surrogate recoveries: Delete the phrase: The limits for surrogate recoveries are updated biannually (~~see TestAmerica Nashville's current Control Limits Manual (GLM).~~).

Section 10.2, Calibration, Initial Calibration, Steps 1 and 2: Add bold column.

- Prepare calibration standards at five (minimum) different concentrations.

Traditional Volume Concentration (µg/mL)	RVE/LVI Concentration (µg/mL)	µL of 200 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL of 200 µg/mL standard/500 µL (5 µL injection)
2	0.4	5	1

10	2	25	5
20	4	50	10
50	10	125	25
80	16	200	40
100	20	250	50

For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:

Concentration (µg/mL)	RVE/LVI Concentration (µg/mL)	µL of 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL of 10 µg/mL standard/500 µL (5 µL injection)
0.05*	0.01	2.5	0.5
0.1	0.02	5	1
0.5	0.1	25	5
1	0.2	50	10
5	1.0	250	50
10	2.0	500	100

*The lowest calibration 0.05 µg/mL standard must be used for low-level SIM analysis on samples from Wisconsin.

Section 16.0, Method Modification: Add item 5 to the table:

Item	Modification
5	RVE/LVI

<i>Michael A. Dunn</i>	3/11/13	<i>[Signature]</i>	3/11/13
Technical and Quality Assurance Approval	Date	Operations Manager Approval	Date

SOP Number/Revision No.: 8270 / NV/SA04-22.15




Effective Date: 3/29/2013

Last Mod. Date: 12/31/12

SOP Title: **Method 8270C/D: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)**

Revision Number with Mod ID: 15a

**Title: SEMIVOLATILE ORGANIC COMPOUNDS BY GAS
CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
EPA METHOD 8270C/D**

Approvals (Signature/Date)			
	12/28/12		12/28/12
Cory Spry Extractables Operations Manager	Date	Johnny Davis Health & Safety Manager / Coordinator	Date
	12/28/12		
Michael H. Dunn Technical Director Quality Assurance Manager	Date		

Analyze and report by 8270D for Canadian, NJ, NC, OK, SC, and WV samples.

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1.0 Scope and Application

1.1 Analyte, Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of oily wastes, soils/sediments, concrete, and water samples. The following compounds can be determined by this method:

Analyte	CAS #	Analyte	CAS #
Acenaphthene ^{1, 2, 5}	83-32-9	Hexachlorocyclopentadiene ^{1, 2}	77-47-4
Acenaphthene-d ₁₀ (IS)		Hexachloroethane ^{1, 2}	67-72-1
Acenaphthylene ^{1, 2, 5}	208-96-8	Hexachlorophene ²	70-30-4
Acetophenone ²	98-86-2	Hexachloropropene ²	1888-71-7
2-Acetylaminofluorene ²	53-96-3	Indeno(1,2,3-cd)pyrene ^{1, 2, 5}	193-39-5
4-Aminobiphenyl ²	92-67-1	Indene ⁴	
Aniline ²	62-53-3	Isodrin ²	465-73-6
Anthracene ^{1, 2, 4, 5}	120-12-7	Isophorone ^{1, 2}	78-59-1
Aramite ²	140-57-8	cis-Isosafrole ²	17627-76-8
Azobenzene ³	103-33-3	trans-Isosafrole ²	4043-71-4
Benidine ³	92-87-5	Kepone ²	143-50-0
Benzoic acid ³	65-85-0	Methapyrene ²	91-80-5
Benz(a)anthracene ^{1, 2, 4, 5}	56-55-3	3-Methylcholanthrene ²	56-49-5
Benzo(b)fluoranthene ^{1, 2, 4, 5}	205-99-2	6-Methylchrysene ⁴	1705-85-7
Benzo(j)fluoranthene ⁴		4,4'-Methylenebis(2-chloroaniline) (MOC) ³	101-14-4
Benzo(k)fluoranthene ^{1, 2, 4, 5}	207-08-9	Methyl methanesulfonate ²	66-27-3
Benzo(g,h,i)perylene ^{1, 2, 5}	191-24-2	1-Methylnaphthalene ^{3, 4, 5}	90-12-0
Benzo(a)pyrene ^{1, 2, 4, 5}	50-32-8	2-Methylnaphthalene ^{1, 2, 5}	91-57-6
Benzyl alcohol ²	100-51-6	Methyl parathion ²	298-00-0
Bis(2-chloroethoxy)methane ^{1, 2}	111-91-1	2-Methylphenol ^{1, 2, 4}	95-48-7
Bis(2-chloroethyl)ether ^{1, 2}	111-44-4	3-Methylphenol ^{1, 2, 4}	108-39-4
Bis(2-chloroisopropyl)ether ^{1, 2}	108-90-1	4-Methylphenol ^{1, 2, 4}	106-44-5
Bis(2-ethylhexyl)adipate ³	109-23-1	Naphthalene ^{1, 2, 4, 5}	91-20-3
Bis(2-ethylhexyl)phthalate ^{1, 2, 4}	117-81-7	Naphthalene-d ₈ (IS)	
Bisphenol A ³	80-05-7	1,4-Naphthoquinone ²	130-15-4
4-Bromophenyl phenylether ^{1, 2}	101-55-3	1-Naphthylamine ²	134-32-7
Butyl benzyl phthalate ^{1, 2, 4}	85-68-7	2-Naphthylamine ²	91-59-8
Carbazole ¹	86-74-8	2-Nitroaniline ^{1, 2}	88-74-4
4-Chloroaniline ^{1, 2}	106-47-8	3-Nitroaniline ^{1, 2}	99-09-2
Chlorobenzilate ²	510-15-6	4-Nitroaniline ^{1, 2}	100-01-6
4-Chloro-3-methylphenol ^{1, 2}	59-50-7	Nitrobenzene ^{1, 2}	98-95-3
1-Chloronaphthalene ³	90-13-1	Nitrobenzene-d ₅ (surr)	
2-Chloronaphthalene ^{1, 2}	91-58-7	2-Nitrophenol ¹	88-75-5
2-Chlorophenol ^{1, 2}	95-57-8	4-Nitrophenol ^{1, 2}	100-02-7
2-Chlorophenol-d ₄ (surr)		5-Nitro-o-toluidine ²	99-55-8
4-Chlorophenyl phenylether ²	7005-72-3	Nitroquinoline-1-oxide ²	56-57-5
Chrysene ^{1, 2, 4, 5}	218-01-9	n-Nitrosodi-n-butylamine ²	924-16-3
Chrysene-d ₁₂ (IS)		n-Nitrosodiethylamine ²	55-18-5
n-Decane ³	124-18-5	n-Nitrosodimethylamine ²	62-75-9
Diallate (cis and trans) ²	2303-16-4	n-Nitrosomethylethylamine ²	10595-95-6
Dibenz(a,h)acridine ⁴	226-36-8	n-Nitrosodiphenylamine ^{1, 2} and Diphenylamine	86-30-6 and 122-39-4
Dibenz(a,i)acridine ³	224-42-0	n-Nitrosodi-n-propylamine ^{1, 2}	621-64-7
Dibenz(a,h)anthracene ^{1, 2, 4, 5}	53-70-3	n-Nitrosomorpholine ²	59-89-2
Dibenzofuran ^{1, 2}	132-64-9	n-Nitrosopiperidine ²	100-75-4
2,3-Dichloroaniline ³	608-27-5	n-Nitrosopyrrolidine ²	930-55-2

1,2-Dichlorobenzene ^{1, 2, 4}	955-50-1	<i>n-Octadecane</i> ³	593-45-3
<i>1,2-Dichlorobenzene-d₄(surr)</i>		Parathion ²	56-38-2
1,3-Dichlorobenzene ^{1, 2, 4}	541-73-1	Pentachlorobenzene ²	608-93-5
1,4-Dichlorobenzene ^{1, 2, 4}	106-46-7	<i>Pentachloroethane</i> ²	76-01-7
1,4-Dichlorobenzene-d ₄ (IS)		Pentachloronitrobenzene ²	82-68-8
3,3'-Dichlorobenzidine ^{1, 2}	91-94-1	Pentachlorophenol ^{1, 2}	87-86-5
2,4-Dichlorophenol ^{1, 2}	120-83-2	Perylene-d ₁₂ (IS)	
2,6-Dichlorophenol ²	87-65-0	Phenacetin ²	62-44-2
Diethyl phthalate ^{1, 2, 4}	84-66-2	Phenanthrene ^{1, 2, 4, 5}	85-01-8
Dimethoate ²	60-51-5	Phenanthrene-d ₁₀ (IS)	
Dimethylaminoazobenzene ²	60-11-7	Phenol ^{1, 2, 4}	108-95-2
7,12-Dimethylbenz(a)anthracene ^{2, 4}	57-97-6	Phenol-d ₅ (surr)	
3,3'-Dimethylbenzidine ²	119-93-7	1,4-Phenylenediamine	106-50-3
2,4-Dimethylphenol ^{1, 2, 4}	105-67-9	Phorate ²	298-02-2
a,a-Dimethylphenethylamine ²	122-09-8	2-Picoline (2-Methylpyridine) ²	109-06-8
Dimethyl phthalate ^{1, 2, 4}	131-11-3	Pronamide ²	23950-58-5
Di-n-butyl phthalate ^{1, 2, 4}	84-74-2	Pyrene ^{1, 2, 4, 5}	129-00-0
1,3-Dinitrobenzene ²	99-65-0	Pyridine ^{2, 4}	110-86-1
4,6-Dinitro-2-methylphenol ^{1, 2}	534-52-1	<i>Quinoline</i> ⁴	91-22-5
2,4-Dinitrophenol ^{1, 2, 4}	51-28-5	Safron	94-59-7
2,4-Dinitrotoluene ^{1, 2, 5}	121-14-2	Terphenyl-d ₁₄ (surr)	1718-51-0
2,6-Dinitrotoluene ^{1, 2, 5}	606-20-2	<i>Alpha-Terpineol</i> ³	7785-53-7
Dinoseb ²	88-85-7	1,2,4,5-Tetrachlorobenzene ²	95-94-3
<i>1,4-Dioxane</i>	123-91-9	2,3,4,6-Tetrachlorophenol ²	58-90-2
1,2-Diphenylhydrazine ³	122-66-7	<i>Tetraethyl dithiopyrophosphate (Sulfotepp)</i> ²	3689-24-5
Di-n-octyl phthalate ^{1, 2, 4}	117-84-0	Tetraethylpyrophosphate ³	107-49-3
Disulfoton ²	298-04-4	Thionazine ²	297-97-2
Ethyl methanesulfonate ²	62-59-0	Thiophenol ⁴	108-98-5
Famphur ³	54-85-7	o-Toluidine ²	95-53-4
Fluoranthene ^{1, 2, 4, 5}	206-44-0	2,4,6-Tribromophenol (surr)	118-79-6
Fluorene ^{1, 2, 5}	86-73-7	1,2,4-Trichlorobenzene ^{1, 2}	120-82-1
2-Fluorobiphenyl(surr)	321-60-8	2,4,5-Trichlorophenol ^{1, 2}	95-95-4
2-Fluorophenol (surr)	367-12-4	2,4,6-Trichlorophenol ^{1, 2}	88-06-2
Hexachlorobenzene ^{1, 2}	118-74-1	o,o,o-Triethylphosphorothioate ²	126-68-1
Hexachlorobutadiene ¹	87-68-3	1,3,5-Trinitrobenzene ²	99-35-4
<i>Compounds in italics are not present in the EPA method.</i>			
¹ – Normal laboratory 8270 compounds. For OH VAP projects, report only compounds designated by a 1 superscript; see Attachment 5.			
² - Appendix IX compounds (by request only)			
³ - additional compounds available by this method (by request only)			
⁴ - Skinner List for Refinery Waste compounds (by request only)			
⁵ - Compounds that are available by GC/MS-SIM (by request only)			
IS = These compounds are used as internal standards.			
surr = These compounds are used as surrogates.			

This method is used to quantitate neutral, acidic, and basic organic compounds that are soluble in Methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica capillary column coated with a slightly polar methyl silicone phase. This method is not appropriate for the quantitation of multi-component analytes, e. g., Aroclors, Toxaphene, Chlordane, etc., because of limited sensitivity for those analyses. This method is appropriate for confirmation of the presence of these analytes when concentration in the extract

permits. However, it is appropriate for the multi-component analyte, Diesel Range Organics (DRO), as requested by Missouri and California; see Attachment 6.

1.2 Reporting Limits: The laboratory typical report limit (RL) is approximately 2 - 100 µg/L for water samples, 67 - 670 µg/kg (wet weight) for soil/sediment samples, and 10 - 1000 mg/kg for wastes (dependent on matrix and method of preparation). See the following table for typical RLs for each compound. For the most current analyte RLs, refer to LIMS.

Typical Reporting Limits for 8270 Compounds

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦Acenaphthene	10	0.333	♦Isosafrole	50	1.67
♦Acenaphthylene	10	0.333	♦Kepone	10	0.333
♦Acetophenone	10	0.333	♦Methapyrilene	50	0.333
♦2-Acetylaminofluorene	10	0.333	♦3-Methylcholanthrene	10	0.333
♦4-Aminobiphenyl	10	0.333	♦6-Methylchrysene	10	0.333
♦Aniline	10	0.333	♦Methyl methanesulfonate	10	0.333
♦♦Anthracene	10	0.333	♦1-Methylnaphthalene	10	0.333
♦Aramite	50	1.67	♦2-Methylnaphthalene	10	0.333
Atrazine	10	0.333	♦Methylparathion	10	1.67
Azobenzene	10	0.333	♦♦3-Methylphenol	10	0.333
Benzaldehyde	10	1.67	♦♦3,4-Methylphenol	10	0.333
Benzidine	100	1.67	♦♦Naphthalene	10	0.333
Benzoic acid	50	1.67	♦1,4-Naphthoquinone	10	1.67
♦♦Benzo(a)anthracene	10	0.333	♦1-Naphthylamine	10	0.333
♦♦Benzo(a)pyrene	10	0.333	♦2-Naphthylamine	10	0.333
♦♦Benzo(b)fluoranthene	10	0.333	♦2-Nitroaniline	25	0.833
♦Benzo(g,h,i)perylene	10	0.333	♦3-Nitroaniline	25	0.833
♦Benzo(j)fluoranthene	10	0.333	♦4-Nitroaniline	25	0.833
♦♦Benzo(k)fluoranthene	10	0.333	♦Nitrobenzene	10	0.333
♦Benzyl alcohol	10	0.333	♦5-Nitro-o-toluidine	10	1.67
Biphenyl	10	0.333	♦2-Nitrophenol	10	0.333
♦Bis(2-chloroethoxy) methane	10	0.333	♦♦4-Nitrophenol	25	0.833
♦Bis(2-chloroethyl) ether	10	0.333	♦Nitroquinoline-1-oxide	10	0.333
♦Bis(2-chloroisopropyl) ether	10	0.333	♦n-Nitrosodiethylamine	10	0.333
♦♦Bis(2-ethylhexyl) phthalate	10	0.333	♦n-Nitroso-dimethylamine	10	0.333
♦4-Bromophenylphenyl ether	10	0.333	♦n-Nitrosodi-n-butylamine	10	1.67
♦♦Butyl benzyl phthalate	10	0.333	♦n-Nitroso-di-n-propylamine	10	0.333
Caprolactum	10	0.333	♦n-Nitroso-diphenylamine and Diphenylamine	10	0.333
Carbazole	10	0.333	♦n-Nitrosomethylethylamine	10	0.333
♦4-Chloro-3-methylphenol	10	0.333	♦n-Nitrosomorpholine	10	1.67
♦4-Chloroaniline	10	0.333	♦n-Nitrosopiperidine	10	1.67
♦Chlorobenzilate	10	0.333	♦n-Nitrosopyrrolidine	10	1.67
1-Chloronaphthalene	10	0.333	Octadecane	50	0.333

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦2-Chloronaphthalene	10	0.333	♦Parathion	10	1.67
♦2-Chlorophenol	10	0.333	♦Pentachlorobenzene	10	1.67
♦4-Chlorophenylphenyl ether	10	0.333	♦Pentachloroethane	10	0.333
♦♣Chrysene	10	0.333	♦Pentachloronitrobenzene	10	1.67
♦cis-Diallate	10	0.333	♦Pentachlorophenol	25	0.833
♦trans-Diallate	10	0.333	♦Phenacetin	10	1.67
♦Dibenzofuran	10	0.333	♦♣Phenanthrene	10	0.333
♣Dibenz(a,h)acridine	10	0.333	♦♣Phenol	10	0.333
Dibenz(a,i)acridine	10	0.333	♦1,4-Phenylenediamine	50	0.333
♦♣Dibenzo(a,h)anthracene	10	0.333	♦Phorate	10	0.333
♦♣1,2-Dichlorobenzene	10	0.333	♦2-Picoline	10	0.333
♦♣1,3-Dichlorobenzene	10	0.333	♦Pronamide	10	1.67
♦♣1,4-Dichlorobenzene	10	0.333	♦♣Pyrene	10	0.333
♦3,3'-Dichlorobenzidine	10	0.333	♦♣Pyridine	10	0.67
♦2,4-Dichlorophenol	10	0.333	♣Quinoline	10	0.333
♦2,6-Dichlorophenol	20	0.333	♦Sairfos	10	0.333
3,4-Dichlorophenol	10	0.333	♦Terbufos	50	167
♦♣Diethyl phthalate	10	0.333	♦1,2,4,5-Tetrachlorobenzene	10	1.67
♦Dimethoate	10	1.67	♦2,3,4,6-Tetrachlorophenol	10	0.333
♦p-Dimethylaminoazobenzene	10	1.67	♦Tetraethylpyrophosphate, Sulfotep	10	1.67
♦3,3'-Dimethylbenzidine	50	0.333	♦Thionazine	10	1.67
♦♣7,12-Dimethylbenz(a)anthracene	10	0.333	♣Thiophenol	50	1.67
♦a,a-Dimethylphenethylamine	50	1.67	♦o-Toluidine	10	1.67
♦♣2,4-Dimethylphenol	10	0.333	♦1,2,4-Trichlorobenzene	10	0.333
♦♣Dimethyl phthalate	10	0.333	♦2,4,5-Trichlorophenol	10	0.667
♦♣Di-n-butyl phthalate	10	0.333	♦2,4,6-Trichlorophenol	10	0.333
♦1,3-Dinitrobenzene	10	1.67	♦o,o,o-Triethylphosphorothioate	10	1.67
♦4,6-Dinitro-2-methylphenol	25	0.833	♦1,3,5-Trinitrobenzene	10	0.333
♦♣2,4-Dinitrophenol	25	0.833	Acenaphthene, SIM	0.10	0.00333
♦2,4-Dinitrotoluene	10	0.333	Acenaphthylene, SIM	0.10	0.00333
♦2,6-Dinitrotoluene	10	0.333	Anthracene, SIM	0.10	0.00333
♦♣Di-n-octyl phthalate	10	0.333	Benzo(a)anthracene, SIM	0.10	0.00333
♦Dinoseb	10	0.333	Benzo(a)pyrene, SIM	0.10	0.00333
1,4-Dioxane	10	0.333	Benzo(b)fluoranthene, SIM	0.10	0.00333
1,2-Diphenylhydrazine	10	0.333	Benzo(g,h,i)perylene, SIM	0.10	0.00333
♦Disulfoton	10	1.67	Benzo(k)fluoranthene, SIM	0.10	0.00333
♦Ethyl methanesulfonate	10	0.333	Chrysene, SIM	0.10	0.00333
♦Famphur	10	0.333	Dibenzo(a,h)anthracene, SIM	0.10	0.00333
♦♣Fluoranthene	10	0.333	2,4-Dinitrotoluene, SIM	0.2	0.0067

Analyte	Water RL µg/L	Soil RL mg/kg	Analyte	Water RL µg/L	Soil RL mg/kg
♦ Fluorene	10	0.333	2,6-Dinitrotoluene, SIM	0.2	0.0067
♦ Hexachlorobenzene	10	0.333	Fluoranthene, SIM	0.10	0.00333
♦ Hexachlorobutadiene	10	0.333	Fluorene, SIM	0.10	0.00333
♦ Hexachlorocyclopentadiene	10	0.333	Indeno(1,2,3-cd)pyrene, SIM	0.10	0.00333
♦ Hexachloroethane	10	0.333	1-Methylnaphthalene, SIM	0.10	0.00333
♦ Hexachlorophene	50	3.33	2-Methylnaphthalene, SIM	0.10	0.00333
♦ Hexachloropropene	50	3.33	Naphthalene, SIM	0.10	0.00333
♦ Indeno(1,2,3-c,d)pyrene	10	0.333	Phenanthrene, SIM	0.10	0.00333
♣ Indene	10	1.67	Pyrene, SIM	0.10	0.00333
♦ Isodrin	10	0.333	California / Missouri ORO	500	20
♦ Isophorone	10	0.333	California / Missouri ORO	500	20

♦ indicates Appendix IX compound

♣ Skinner List compound

Bold compounds are reported in a standard list.*Italicized compounds are only available upon special request by this method.*

SIM = Selective Ion Monitoring

1.3 The following compounds may require special treatment when being determined by this method:

- Benzidine may be subject to oxidative losses during solvent concentration, and its chromatographic behavior is poor.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- n-Nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- n-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Pyridine may perform poorly at the GC injection port temperatures listed in the method. Lowering the injection port temperature may reduce the amount of degradation. Use caution if modifying the injection port temperature as the performance of other analytes may be adversely affected.

1.4 If for any reason a part of this method cannot be followed, seek the guidance of the Department Supervisor or the Technical Director. All abnormalities must be noted on the data or the benchsheet and in the Laboratory Information Management System (LIMS).

2.0 Summary of Method

2.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation. See SOPs 3510 / NV03-24 for waters, 3550 / NV03-23 and 3541 / NV03-231 for soils and concrete, and 3580 / NV03-106 for oils, and, if necessary, sample cleanup procedures.

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass

spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using at least a multi-point calibration curve.

3.0 Definitions

3.1 Reduced Volume Extraction / Large Volume Injection (RVE/LVI): The option to use a reduced sample volume for extraction combined with a larger volume extract injection on the instrument. Volumes for this option are shown in this document as RVE/LVI in brackets.

3.2 See TestAmerica Nashville's Quality Assurance Manual Appendix 5 for laboratory definitions. Also, refer to Controlled Document QAF-45, TestAmerica Nashville Acronyms, Keywords, and Definitions.

4.0 Interferences

Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. This method may involve hazardous material, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements:

- The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. Be aware of the locations of those zones, and cool them to room temperature prior to working on them.
- The mass spectrometer is under high vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used: The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatography/mass spectrometer/data system
 - Gas chromatograph (HP or Agilent): Analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.
 - Column: 30 m x 0.25 mm ID with a 0.25 μ m film thickness silicone-coated fused-silica capillary column (Phenomenex ZB-5, or equivalent) [RVE/LVI: and a 5 m x 0.32 mm ID guard column (Phenomenex 7CG-G000-000-GZC, or equivalent)].
 - Mass spectrometer capable of scanning from 35 to 500 amu every 1 second less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for Decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 2 when 1 μ L of the GC/MS tuning standard is injected (50 ng or less of DFTPP).
 - Data system (Chemstation with Enviroquant): A computer system is interfaced to the mass spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software is also available that allows integrating the abundances in any EICP between specified time or scan-number limits. The EPA/NIST Mass Spectral Library is also available.
 - Suggested operating conditions (may vary by instrument; see maintenance log for current program):

Mass range:	35-500 amu
Scan time:	1 second/scan
Initial temperature:	40°C hold for 2 minutes
Temperature program:	Rate 1: 15°C/minute to 160°C
	Rate 2: 10°C/minute to 320°C
Final temperature:	320°C hold for at least 1.5 minute.
Injector temperature:	240-250°C
Transfer line temperature:	280°C
Source temperature:	According to manufacturer's specifications (nominally 250 – 275°C)
Injector:	Grob-type, split-less
Injection volume:	1 μ L [RVE/LVI: 5 μ L]
Carrier gas:	Helium at 1 mL/minute

6.2 Supplies

- Microsyringe, 10 µL.
- Balance, analytical, capable of weighing 0.0001 g
- Glass vials, glass with PTFE (polytetrafluoroethylene)-lined screw-caps or crimp tops.
- Volumetric flasks, Class A, appropriate sizes with ground-glass stoppers.

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Reagent water, analyte-free.

7.3 Stock Calibration Standards: Commercially prepared, certified stock standards are purchased:

- The primary standard for the typical 8270 compound list is from Ultra Scientific CUS-6150, or equivalent, with the required targets at 200 µg/mL.
- For PAHs by SIM, use Accustandard Z-014G-FL, or equivalent, with the target PAHs at 2000 µg/mL.
- For Appendix IX and miscellaneous compounds, primary source standards are purchased from NSI; equivalent substitutes are acceptable.

Analyte/Analyte Group	NSI Catalog Number	Concentration (µg/mL)
AIX Mix	C-426	2000
Acid Extractables II	C-415	2000
Amines	C-412	2000
Aramite	922-05-02	2000
a,a-Dimethylphenylamine	922-05-02	2000
Benzidines	C-411	2000
BNA II mix	C-413	2000
B/N III mix	C-414	2000
Hexachlorophene	323-03	5000
Sulfonates	C-416	2000
8270 OP Pest	C-417	2000

7.4 Matrix Spike and Laboratory Control Standard contains all targets to be reported on the samples. The same compounds mentioned in Section 7.3 are designated as the SPCCs and CCCs for 8270C.

- For both a long semivolatile list and the PAH list by SIM, purchase as the second source a 100 µg/mL standard, NSI Catalog # c-408-50x, or equivalent.
- For Appendix IX and miscellaneous compounds, these second source standards are acceptable, as well as equivalents:

Analyte/Analyte Group	Restek Catalog Number	Concentration (µg/mL)
AIX #1 Mix	31625	2000
AIX #2 Mix	31806	2000
Calibration Mix	31618	2000
OP Mix	32419	2000

7.5 Internal standard solutions: The internal standards are 1,4-Dichlorobenzene-d₄, Naphthalene-d₈, Acenaphthene-d₁₀, Phenanthrene-d₁₀, Chrysene-d₁₂, and Perylene-d₁₂.

- Purchase certified, internal standard at 4000 µg/mL, NSI C-394, or equivalent.

7.6 GC/MS Tuning Standard: A Methylene chloride solution containing 50 µg/mL of Decafluorotriphenylphosphine (DFTPP) is prepared. The standard also contains 50 µg/mL each of 4, 4'-DDT, Pentachlorophenol, and Benzidine to verify injection port inertness and GC column performance.

- Purchase the tuning standard at 1000 µg/mL from Ultra Scientific, Catalog GCM-150, or equivalent.

7.7 Surrogate standards: The surrogates are Phenol-d₅, 2-Fluorophenol, 2,4,6-Tribromophenol, Nitrobenzene-d₅, 2-Fluorobiphenyl, and p-Terphenyl-d₁₄.

- Purchase the acid/base/neutral and PAH SIM surrogates from NSI CUS-7070, or equivalent, at 50 µg/mL.

7.8 Acetone, Hexane, Methylene chloride, Isooctane, Carbon disulfide, Toluene, and other appropriate solvents, commercial source.

7.9 Sodium sulfate for blank and LCS soil matrix.

7.10 Transfer the stock standard solutions into bottles with PTFE-lined screw-caps. Store, protected from light, at -10°C or less or as recommended by the standard manufacturer. Stock standard solutions must be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Replace after **one year or sooner** if comparison with quality control check samples indicates a problem, or if the vendor specifies an expiration date sooner than one year.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	3 L, amber glass with Teflon®-lined cap	1 L [RVE/LV: 250 mL]	Cool 0-6°C. Keep in dark.	7 days from collection until extraction, 40 days after extraction	SW-846 Chapter 2
Soil, Oil, Concrete	4 oz. glass jar with Teflon®-lined cap	50 g	Cool 0-6°C.	14 days from collection until extraction, 40 days after extraction	

9.0 Quality Control

The laboratory maintains a formal quality assurance program and records to document the quality of the data generated.

Certain quality control and reporting criteria may vary depending on whether SW-846 8000B or 8000C criteria are required. In these cases, both sets of criteria have been noted in this SOP. 8000C criteria are required to be applied ONLY to Arizona and Washington samples. All other samples must be processed against referenced 8000B criteria. Exceptions may be required on a project-specific basis.

9.1 Sample QC:

The following QC samples are run with each batch of no more than 20 samples.			
QC Check	Frequency	Acceptance Criteria ¹	Corrective Action ²
Method blank	One per analytical prep batch	No analytes detected $\geq \frac{1}{2}$ RL or MDL, whichever is greater	Correct problem then re-prepare ³ and analyze method blank and all samples processed with the contaminated blank.
LCS ⁶ for all analytes (2 nd source) ⁶	One ⁶ per prep batch	See LIMS and footnote 4 below.	Correct problem then re-prepare ⁴ and analyze the LCS and all samples in the affected analytical batch. ⁴ If high and target is ND, OK to report.
MS/MSD (2 nd source)	One per batch per matrix, if insufficient sample for MS/MSD, qualify data ³	See LIMS.	None (the LCS is used to evaluate to determine if the batch is acceptable).
Surrogate(s)	Every sample, spike, standard, and blank	See LIMS. ⁵	Check system, re-analyze, re-prepare ^{3,5} .

¹This is a summary of the acceptance criteria.

²All abnormalities must be noted on the data, the benchsheet and in LIMS.

³If unable to re-prepare samples because of insufficient sample volume or the holding time has expired, then place a comment on the benchsheet and in LIMS.

⁴If the LCS exceeds the upper control limit AND a sample from that batch is greater than the RL, re-prepare and re-analyze the batch. If the LCS exceeds the upper control limit AND the samples from that batch is less than the RL, the data is acceptable to report.

⁵If the surrogate % recovery exceeds the upper control limit AND a sample result is positive above the RL, re-prepare and re-analyze the batch. If the surrogate % recovery exceeds the upper control limit AND the sample is less than the RL, data is acceptable to report. If the surrogate % recovery is lower than the lower control limit, re-prepare the sample. OH VAP requires all surrogates to be in control; otherwise, the samples must be re-prepared and re-analyzed.

⁶LCSD is required for AZ, MA, TX, WV.

- A **Method blank** is extracted with every batch of samples.
- A **Laboratory Control Sample (LCS)** is included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume (reagent water for water batches, Sodium sulfate for soil batches). It is spiked with the same analytes at the same concentrations as the matrix spike. **All target analytes must meet the LCS QC criteria** (laboratory historical limits in LIMS). However, if the LCS is high, and a target is ND, it is acceptable to report the result.
 - The LCS spike is from a **different source than the calibration standards**. Using the 100 µg/mL LCS/MS/MSD standard:
 - **For Non-SIM batches:**
 - Water: add 500 µL [RVE/LVI: 100 µL] of the standard per liter reagent water before extraction by Method 3510C.
 - Soil: add 500 µL of the standard per 30 gram Sodium sulfate before extraction.
 - TCLP: add 1 mL [RVE/LVI: 200 µL] of the standard/500 mL TCLP extraction fluid before extraction by Method 3510C.
 - The final concentration is 50 µg/mL on column.
 - **For SIM batches:**
 - Water: add 1 mL [RVE/LVI: 200 µL] of a 100 X dilution of the NSI standard per liter reagent water.
 - Soil: add 1 mL of a 100 X dilution of the NSI standard per 30 g Sodium sulfate.
 - The final concentration in the extracts is 1.0 µg/mL.
 - **Matrix Spike / Matrix Spike Duplicate:** Documenting the effect of the matrix includes the analysis of at least one matrix spike/matrix spike duplicate pair.
 - The MS/MSD spike is from a **different source** than the calibration standards. Using the 100 µg/mL LCS/MS/MSD standard:
 - **For Non-SIM batches:**

- Water: add 500 μL [RVE/LVI: 100 μL] of the standard per liter client sample.
- Soil: add 500 μL of the standard to 30 g client sample.
- TCLP: add 1 mL [RVE/LVI: 200 μL] of the standard per 500 mL client TCLP extract.
- The final concentration is 50.0 $\mu\text{g/mL}$ on column..
- **For SIM batches:**
 - Water: add 1 mL [RVE/LVI: 200 μL] of a 100 X dilution of the NSI standard per liter reagent water.
 - Soil: add 1 mL of 100 X dilution per 30 g client sample.
 - The final concentration is 1.0 $\mu\text{g/mL}$ on column.
- **Surrogate recoveries:** The laboratory evaluates surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The limits for surrogate recoveries are updated biannually (see TestAmerica Nashville's current Control Limits Manual (CLM)). If any surrogate is outside QC limits, and there is no obvious matrix interference, then re-analyze and/or re-extract the sample. If surrogates are still outside limits, flag the data in LIMS. However, if high and all results are non detect, results are reportable. If surrogate recoveries are low, re-prepare the batch.
- **For Non-SIM,** add 1000 μL [RVE/LVI: 200 μL] of the surrogate standard at a concentration of 50 $\mu\text{g/mL}$ to each sample and batch QC samples prior to extraction for a 50 $\mu\text{g/mL}$ concentration.
- **For SIM,** prepare a 1 $\mu\text{g/mL}$ standard (500 μL surrogate standard) to 500 mL in methanol. Add 1.0 mL [RVE/LVI: 200 μL] to samples and QC (blanks, MS/MSD and LCS) prior to extraction. The concentration is 1.0 $\mu\text{g/mL}$.

9.2 Instrument QC

QC Check	Frequency	Acceptance Criteria ²	Corrective Action ³									
GC/MS Tuning												
a. Check of mass spectral ion intensities ¹ , i.e., Tune	Prior to initial calibration or Continuing calibration verification, every 12 hours.	See below in this section for GC/MS Tuning criteria.	Retune the instrument and verify (instrument maintenance may be needed).									
b. Column Breakdown	Prior to initial calibration or Continuing calibration verification, every 12 hours.	Breakdown ratio ≤ 20% (30% for 8270C).	Injector or column maintenance and re-calibration.									
c. Tailing Factor	Prior to initial calibration or Continuing calibration verification, every 12 hours.	<table><tr><td></td><td>8270C</td><td>8270D</td></tr><tr><td>Benzidine</td><td>3</td><td>2</td></tr><tr><td>Pentachlorophenol</td><td>5</td><td>2</td></tr></table>		8270C	8270D	Benzidine	3	2	Pentachlorophenol	5	2	Injector or column maintenance and re-calibration.
	8270C	8270D										
Benzidine	3	2										
Pentachlorophenol	5	2										
Minimum five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	8270C: SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and all other target analytes %RSD for RF ≤ 15% If %RSD is > 15%, linear regression $r^2 \geq 0.990$, $r \geq 0.995$. 8270D: The minimum RF for all compounds in Attachment 5 must be met ⁵ . All targets RSD ≤ 20% or use linear regression.	Correct problem then repeat initial calibration.									

QC Check	Frequency	Acceptance Criteria ²	Corrective Action ³
Initial calibration verification (ICV) must be from a 2 nd source.	Immediately following five-point initial calibration.	All analytes within 30% of expected value.	Correct problem then repeat initial calibration.
Initial calibration blank	Immediately after ICV	All analytes < MDL	Correct problem, re-calibrate.
Continuing calibration verification (CCV)	Daily, before sample analysis and every 12 hours of analysis time.	8270C: SPCCs average RF \geq 0.050 and CCCs: \leq 30% difference (when using RFs) or drift (when using least squares regression). Non-CCC < 20% true; up to 4 may be < 40%. 8270D: The minimum RF for all compounds listed in Attachment 4 must be met and the percent difference or drift for each target compound \leq 20%.	Correct problem then repeat initial calibration and re-analyze all samples since last successful CCV.
Internal Standards	Every sample/standard and blank.	Retention time \pm 30 seconds from retention time of the mid-point std. in the ICAL for CCV. EICP area within -50% to +100% of ICAL mid-point std for the CCV and -50% to +100% of the prior CCV for the samples. See footnote 4 below.	Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning (dilution of the sample may be required, see the supervisor or the technical manager for advice).
Relative Retention Time Window	Each sample.	Relative retention time (RRT) of the analyte within 0.06 RRT units of the RRT of the internal standard.	Correct problem then reprocess or re-analyze all samples analyzed since the last retention time check.
MDL verification (extracted)	Minimum yearly.	Detectible	Re-evaluate MDL standard used and MDL; see the technical manager.

¹8270 requires DFTPP.

²This is a summary of the acceptance criteria.

³All abnormalities must be noted on the data, the benchsheet and in LIMS.

⁴Target compounds associated with failed internal standards must be re-analyzed (undiluted if possible) if additional sample is available; if not available, qualify data in LIMS.

⁵LLCV: If RF is not met at the low-level standard, the criterion for a passing LLCV is detection only and must be run following the CCV.

• Tuning GC/MS Tuning (Full Scan)

- Prior to the analysis of samples or calibration standards, the GC/MS system is hardware-tuned using a 50 ng or less injection of DFTPP (in the GC/MS Tuning Standard).
- The 50 μ g/mL standard is prepared by adding 2.8 mL of 1000 μ g/mL stock standard to 56 mL Methylene chloride. [RVE/LVI: Use a 5X dilution of this solution.]
- Analyses **must** not begin until the tuning criteria are met, and these criteria must be demonstrated at the beginning of each 12-hour shift. Three options are available for acquiring the spectra for reference to meet the DFTPP tuning requirements:

Option 1 It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at the apex \pm 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction **cannot** include any part of the target peak. Sometimes the instrument does

not always correctly identify the apex on some peaks when the peak is not perfectly shaped. It is acceptable to manually identify and average the apex peak ± 1 scan and background correct

Option 2 The entire peak may be averaged and background-corrected. Average scans from 0.1 minute before to 0.1 minute after peak.

Option 3 A single scan at the apex (only) may also be used for the evaluation of the tune. Background correction is required.

Note: It is acceptable to adjust parameters within the specifications set by the manufacturer or the analytical method to properly tune the instrument. If the tune verification does not pass, it may be necessary to clean the source or perform additional maintenance. Document any maintenance in the instrument log. **Excessive adjusting (more than two tries) without clear documentation is not allowed. No more than two consecutive tunes may be attempted. Perform necessary maintenance.**

- All subsequent standards, samples, controls, and blanks associated with a DFTPP tune must use the identical mass spectrometer instrument conditions.
- Use the DFTPP mass intensity criteria as follows as tuning acceptance criteria.

DFTPP Key Ions and Ion Abundance Criteria

Mass	m/z Abundance criteria
51	30-60 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40-60 percent of mass 198.
197	Less than 1 percent of mass 198.
198	Base peak, 100 percent relative abundance.
199	5-9 percent of mass 198.
275	10-30 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than mass 443.
442	Greater than 40 percent of mass 198.
443	17-23 percent of mass 442.

- **Breakdown Standard:** The GC/MS Tuning Standard is also used to assess the injection port inertness by evaluating the degradation of DDT to DDE and DDD. This ratio must **not** exceed 20%; see Section 9.2 for **percent breakdown** calculation. Perform injector or column maintenance and recalibrate if the ratio maximum is exceeded for either compound. The breakdown of DDT is measured **before** verification standards and samples are analyzed and every 12 hours throughout the sequence.
- **Tailing Factor:** To evaluate the GC column, Benzidine and Pentachlorophenol (in the GC/MS Tuning Standard) must be present at their normal responses and evaluated for peak tailing. The Benzidine and Pentachlorophenol tailing factors are calculated by the following equation:

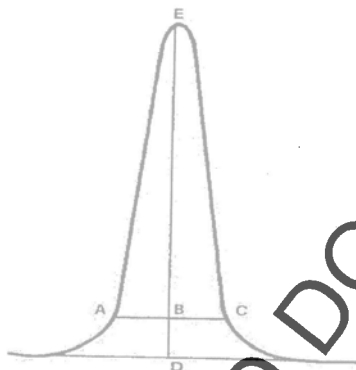
$$\text{Tailing factor} = \text{BC/AB}$$

Maximum Tailing Factor Ratios

Tailing Factor Compounds	8270C	8270D
Benzidine	3	2
Pentachlorophenol	5	2

where the peak is defined as follows: AC is the width at 10% height. DE is the height of peak and B is the height at 10% DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

FIGURE 1
TAILING FACTOR CALCULATION



TAILING FACTOR = $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11} = 1.1$

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If all of the specified criteria are met, generate a hardcopy of the spectrum, the mass abundance data and the parameters under which the scans were acquired. This data is filed in the batch for documentation.

GC/MS Tuning (SIM)

- The objective of tuning for conventional full scan analysis is to produce a balanced mass spectrum over the range of interest. The DFTPP tune is, by necessity, done in the full scan mode. However, because the instrument is then immediately switched to the SIM mode, the DFTPP results have limited quality control value. In short, the DFTPP is not analyzed under the same conditions as the calibration, QC, and field samples. In the case of Selective Ion Monitoring (SIM) analysis, there are no comparisons between spectra; instead the instrument is optimized for the relative intensities of the pre-selected analyte ions of interest. For SIM analysis, the laboratory prints out a copy of the autotune (PFTBA) prior to analysis to demonstrate good mass assignment and peak width. No BFB tune is possible while in SIM mode. A printout of the instrument autotune (PFTBA) is included with the data for each day that SIM analyses are run in order to demonstrate good mass assignment and peak width.

- **Calibration:** See Section 10.2.
- **Initial Calibration System Performance Check Compounds (SPCCs):** A system performance check is performed to ensure that minimum average RFs are met before the calibration curve is used.
 - **For 8270C:** The SPCCs are

System Performance Check Standards (SPCCs)	
Base/Neutral Fraction	Acid Fraction
n-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

The **minimum acceptable average RF for the SPCCs is 0.050**. They typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

- **For 8270D**, see Attachment 4 for required minimum response factor criteria for target analytes.
- If the minimum response factors are not met, the system must be evaluated, and corrective action is taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. **This check must be met before sample analysis begins.** An option is to run a LLCCV to show sensitivity.
- **Initial Calibration Calibration Check Compounds (CCCs) for 8270C only:** The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes. The CCCs are:

Calibration Check Compounds (CCC)	
Base/Neutral Fraction	Acid Fraction
Acenaphthene*	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
D,n-octyl phthalate	Pentachlorophenol
Fluoranthene*	2,4,6-Trichlorophenol
Benzo(a)pyrene*	

*For PAH SIM standard

- Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte.

Initial Calibration RSD Differences	
8270C	8270D
The RSD must be less than or equal to 15% for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. However, the RSD for each individual CCC must be less than or equal to 30%.	The RSD must be less than or equal to 20% for each target analyte; if not, see the section on linearity of target analytes in Section 10.2. If not, check

If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

errors in standard preparation, the possible presence of active sites in the GC system, poor chromatographic behaviors for analytes.

- The **Initial Calibration Verification (ICV)** is a second-source standard run immediately after the initial calibration. The acceptance limits are **70-130%** recovery.
 - Add 250 μL of the second-source standard to 250 μL Methylene chloride in an amber vial to prepare an ICV standard at 50 $\mu\text{g/mL}$.
 - For PAHs by SIM, use the second-source standard with the target PAHs at 2000 $\mu\text{g/mL}$. A 10 $\mu\text{g/mL}$ intermediate is made by taking 50 μL of the stock standard along with 20 μL of the base/neutral surrogates. The ICV at 1 $\mu\text{g/mL}$ is made by taking 50 μL of intermediate into 450 μL of Methylene chloride in an amber vial.
 - If ICV acceptance criterion is not met, correct the problem and re-calibrate.
- **Initial Calibration Blank:** a reagent/solvent blank analyzed after the ICV to ensure the system is free of contaminants ($< \text{MDL}$). If not contaminant-free, re-run and/or perform system maintenance.
- The **Continuing Calibration Verification standard (CCV)** is evaluated each day (or every 12 hours) that analysis is performed to determine if the chromatographic system is operating properly.
 - Prepare a daily CCV at 50 $\mu\text{g/mL}$ by adding 100 μL of the primary stock solution to 300 μL Methylene chloride in an amber vial. [RVE/LVI: 20 μL to a final volume of 400 μL Methylene chloride].
 - For PAHs by SIM, use the primary stock standard with the target PAHs at 2000 $\mu\text{g/mL}$. A 10 $\mu\text{g/mL}$ intermediate is made by taking 50 μL of the stock standard along with 20 μL of the base/neutral surrogates. A daily CCV at 1 $\mu\text{g/mL}$ is made by taking 50 μL of intermediate into 450 μL of Methylene chloride in an amber vial. [RVE/LVI: 5 μL to a final volume of 500 μL of Methylene Chloride].
 - The calibration verification standard is prepared at least weekly and stored at 4°C or less.
 - For 8270C, each **SPCC** in the calibration verification (CCV) standard must meet a **minimum response factor of 0.050**. For 8270D, see Attachment 4 for required minimum response factor criteria for target analytes.
 - After the system performance check is met, the **CCCs** are used for 8270C only to check the ongoing validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

CCC % Difference Evaluation Criteria	
8270C	8270D
CCCs $\leq 30\%$ and all other target compounds require an RF $\leq 20\%$; however, up to 5 non-CCC target compounds may be $\leq 40\%$.	If the percent difference for each target compound is less than or equal to 20% , then the initial calibration is assumed to be valid. If the criterion is not met (i. e., greater than 20% difference or drift) for any target, then corrective action is taken prior to the analysis of samples. All targets are considered as CCCs.

- If the CCV criteria cannot be met, a new initial calibration must be generated.
- **Continuing Calibration Blank (CCB):** The CCB is run after each CCV. If the result is not $\leq \text{MDL}$ or $\frac{1}{2} \text{RL}$, correct the problem and re-run.
- **Internal standards** are added to every sample, standard, and QA/QC.

- **Retention time** - The retention times of the internal standards in the continuing calibration verification (CCV) standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than **30 seconds** from that in the mid-point standard level of the **most recent initial calibration** sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- **Response** - If the EICP area for any of the internal standards in the continuing calibration verification (CCV) standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the **most recent initial calibration** sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.
- The laboratory re-analyzes any sample where the internal standard fails and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution.
 - If the internal standard is within criteria, report the second analysis.
 - If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution.
 - If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required.
- The target analytes are quantitated with specific internal standards as shown in this table:

**Semivolatile Internal Standards with
Corresponding Analytes Assigned for Quantitation**

1,4-Dichlorobenzene-d₄	Naphthalene-d₈	Acenaphthene-d₁₀
Aniline	Benzoic acid	Acenaphthene
Benzyl alcohol	Bis(2-chloroethoxy) methane	Acenaphthylene
Bis(2-chloroethyl) ether	4-Chloroaniline	2-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
2-Chlorophenol	2,4-Dichlorophenol	Dibenzofuran
1,3-Dichlorobenzene	2,4-Dimethylphenol	Diethyl phthalate
1,4-Dichlorobenzene	Hexachlorobutadiene	Dimethyl phthalate
1,2-Dichlorobenzene	Isophorone	2,4-Dinitrophenol
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene
Hexachloroethane	Naphthalene	2,6-Dinitrotoluene
2-Methylphenol	Nitrobenzene	Fluorene
3,4-Methylphenol	Nitrobenzene-d ₃ (surr)	2-Fluorobiphenyl (surr)
n-Nitrosodimethylamine	2-Nitrophenol	Hexachlorocyclopentadiene
n-Nitroso-di-n-propyl- amine	1,2,4-Trichlorobenzene	2-Nitroaniline
Phenol	1-Methylnaphthalene	3-Nitroaniline
Phenol-d ₅ (surr)	Hexachloropropene	4-Nitroaniline
Pyridine	2,6-Dichlorophenol	4-Nitrophenol
2-Chlorophenol-d ₄ (surr)	n-Nitrosodi-n-butylamine	2,4,6-Trichlorophenol
1,2-Dichlorobenzene-d ₄ (surr)	1,4-Phenylenediamine	2,4,5-Trichlorophenol
1,4-Dioxane	trans-Isosafrole	1,2-Diphenylhydrazine
Pyridine	1,2,4,5-Tetrachlorobenzene	1,3-Dinitrobenzene
2-Picoline	cis-Isosafrole	Pentachlorobenzene
N-Nitrosomethylethylamine	Safrole	1-Naphthaleneamine
Methyl-methanesulfonate	1-Chloronaphthalene	2-Naphthaleneamine
n-Nitrosodiethylamine	1,4-Naphthoquinone	2,3,4,6-Tetrachlorophenol
Ethylmethanesulfonate	Quinoline	Diphenylamine

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀
Pentachloroethane	Chrysene-d ₁₂	5-Nitro-o-Toluidine
Acetophenone	6-Methylchrysene	trans-Diallate
n-Nitrosopyrrolidine	Dibenz(a,h)acridine	cis-Diallate
2-Toluidine	7,12-Dimethylbenz(a)anthracene	1,3,5-Trinitrobenzene
n-Nitrosomorpholine		Phenacetin
n-Nitrosopiperidine		4-Aminobiphenyl
2-Butoxyethanol		
Indene		
Thiophenol		
Phenanthrene-d ₁₀	Chrysene-d ₁₂	Perylene-d ₁₂
Anthracene	Benzidine	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	Benzo(a)anthracene	Benzo(k)fluoranthene
Di-n-butyl phthalate	Bis(2-ethylhexyl) phthalate	Benzo(g,h)perylene
4,6-Dinitro-2-methylphenol	Butyl benzyl phthalate	Benzo(a)pyrene
Diphenylamine	Chrysene	Dibenz(a,h)anthracene
Fluoranthene	3,3'-Dichlorobenzidine	Di-n-octyl phthalate
Hexachlorobenzene	Pyrene	Indeno(1,2,3-cd)pyrene
n-Nirosodiphenylamine	Terphenyl-d ₁₄ (surr)	Dibenz(a,j)acridine
Pentachlorophenol	4,4'-Methylenebis(2-chloroaniline)	
Phenanthrene	Aramite	
Carbazole	3-Methylcholanthrene	
Bis (2-ethylhexyl)adipate		
Tribromophenol (surr)		
Thionazin		
Pronamide		
Pentachloronitrobenzene		
Dinoseb		
Sulfotepp		
Phorate		
Dimethoate		
Disulfoton		
4-Nitroquinoline-N-oxide		
Methapyrilene		
Isodrin		
Methyl Parathion		
Benzidine		
Parathion		
Hexachlorophene		
Kepone		
4-Dimethylaminozobenzene		
Chlorobenzilate		
3,3'-Dimethylbenzidine		
2-Acetylaminofluorene		

(surr)= surrogate

- The internal standards selected permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation. If interferences are noted, use the next most intense ion as the quantitation ion (i. e., for 1, 4-Dichlorobenzene-d₄, use 152 m/z for quantitation).
- Dilute the 4000 µg/mL internal standard by 2x with Methylene chloride. The resulting

solution contains each internal standard mixture at a concentration of 2000 µg/mL. Each 0.5 mL sample extract undergoing analysis is spiked with 10 µL [RVE/LVI: 2µL] of the internal standard solution, resulting in a concentration of 40 µg/mL of each internal standard.

- For SIM, dilute the 2000 µg/mL internal standard mix by 10x with Methylene chloride for a 200 µg/mL standard. Each 0.5 mL of sample extract undergoing analysis is spiked with 10 µL [RVE/LVI: 5 µL] of internal standard solution, resulting in a concentration of 2 µg/mL of each internal standard.
- Evaluation of target analyte retention time:** The relative retention time (RRT) of each target analyte in each calibration standard must agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement. This criterion is met with the use of a ± 0.25 minute retention time window. Representative retention times are shown in Attachments 1 and 2.
- Method Detection Limit Verification (MDLV):** Annually, verify that the MDL is detectable; if not, re-evaluate the MDL.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Water	1000 mL [RVE/LVI: 250 mL]
Soil, Concrete	30 grams
Oil	1 gram

- Samples are nominally prepared by one of the following methods prior to GC/MS analysis:

Matrix	Methods	SOP #
Water	3510	NV03-24
Soil/sediment/Concrete	3541, 3546, 3550	NV03-231, NV03-25
Oily Waste	3580	NV03-106

- QC samples and client samples must be extracted by the same preparation method.
- All calibration standards, QC samples, and client samples are introduced into the GC/MS using the same injection volume, IS and SS concentrations, and instrument conditions.

10.2 Calibration and Daily Continuing Calibration Verification: Refer to SOP Selection of Calibration Points / CA-T-P-002 and Calibration Curves (General) / CA-Q-S-005. See Section 11 for equations. Calculations are performed by vendor software and LIMS.

- Initially and/or daily, evaluate the DFTPP tune criteria (Section 9.2).
- Evaluate the percent breakdown of DDT (Section 9.2).
- Evaluate the tailing factors for Benzidine and Pentachlorophenol (Section 9.2).

Initial calibration

- Prepare calibration standards at five (minimum) different concentrations.

Concentration (µg/mL)	µL 200 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 200 µg/mL standard/500 µL (5 µL injection)
2	5	1
10	25	5

	<table><tr><td>20</td><td>50</td><td>10</td></tr><tr><td>50</td><td>125</td><td>25</td></tr><tr><td>80</td><td>200</td><td>40</td></tr><tr><td>100</td><td>250</td><td>50</td></tr></table>	20	50	10	50	125	25	80	200	40	100	250	50									
20	50	10																				
50	125	25																				
80	200	40																				
100	250	50																				
	<ul style="list-style-type: none">At least one of the calibration standards corresponds to a sample concentration at or below the laboratory reporting limit (RL). The remaining standards correspond to the working range of the GC/MS system.Each standard contains each analyte to be reported. These target analytes may not include the entire list of analytes for which the method has been demonstrated; however, the laboratory must not report a quantitative result for a target analyte that was not included in the calibration standard(s).Surrogates are included at the same concentrations.The internal standards are at a constant 40 µg/mL. Each 0.5 mL aliquot of calibration standard is spiked with 10 µL [RVE/LVI: 2 µL] of the internal standard solution prior to analysis.																					
2	<p>For SIM, calibration standards are diluted from the intermediate standard solution to give the following concentrations:</p> <table><tr><th>Concentration (µg/mL)</th><th>µL 10 µg/mL standard/500 µL (1 µL injection)</th><th>RVE/LVI: µL 10 µg/mL standard/500 µL (5 µL injection)</th></tr><tr><td>0.05*</td><td>2.5</td><td>0.5</td></tr><tr><td>0.1</td><td>5</td><td>1</td></tr><tr><td>0.5</td><td>25</td><td>5</td></tr><tr><td>1</td><td>50</td><td>10</td></tr><tr><td>5</td><td>250</td><td>50</td></tr><tr><td>10</td><td>500</td><td>100</td></tr></table> <p>*The 0.05 µg/mL standard must be used for low-level SIM analysis on samples from Wisconsin.</p> <ul style="list-style-type: none">Surrogates are included at the same concentrations.The internal standards are at a constant 2 µg/mL.See Attachments 2 and 3 regarding SIM Mass groups.	Concentration (µg/mL)	µL 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 10 µg/mL standard/500 µL (5 µL injection)	0.05*	2.5	0.5	0.1	5	1	0.5	25	5	1	50	10	5	250	50	10	500	100
Concentration (µg/mL)	µL 10 µg/mL standard/500 µL (1 µL injection)	RVE/LVI: µL 10 µg/mL standard/500 µL (5 µL injection)																				
0.05*	2.5	0.5																				
0.1	5	1																				
0.5	25	5																				
1	50	10																				
5	250	50																				
10	500	100																				
3	Analyze 1 µL [RVE/LVI: 5 µL] of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each target analyte. See Attachment 1. Two characteristic ions must be valid for the low standard to be used.																					
4	Calculate response factors (RFs) for each target analyte relative to one of the internal standards.																					
5	Evaluate the system performance check compounds (SPCCs) : The minimum acceptable average RF for these compounds is 0.050 for 8270C. For 8270D, see Attachment 4. This check must be met before sample analysis begins.																					
6	Evaluate the calibration check compounds (CCCs) : If the RSD of any CCC is greater than 8270C criteria, then correct the chromatographic system reactivity before analysis begins. For 8270D, all compounds are treated as CCCs and must be within ± 20%.																					
7	Evaluate the retention times .																					
8	Evaluate the linearity of target analytes - If the RSD (8270C ± 15%; 8270D ± 20%) of any target analytes is within acceptance limits, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor is used for quantitation. If the RSD of any target analyte is greater than the acceptance criteria, linear																					

	regression is used for calibration. The correlation coefficient r^2 must be at least 0.990 ($r \leq 0.995$). If the calibration is not considered linear by either %RSD or linear regression, then correct the problem and re-calibrate. See Section 11 for equations and information on linear regression calibration.
9	Evaluate the intercept; it must be \leq RL or re-calibrate.
10	Evaluate the success of the initial calibration by running an Initial Calibration Verification (ICV).
11	Evaluate the Initial Calibration Blank to be sure it is free of contaminants.

Initial Calibration Sequence Summary

1	DFTPP Tuning Criteria/DDT Breakdown/Tailing Factors
2	Calibration Standards
3	ICV
4	ICB

Daily continuing calibration verification - Calibration verification is performed at the beginning of each 12-hour analytical shift.	
1	The initial calibration for each compound of interest is verified once every 12 hours and prior to sample analysis by analyzing a continuing calibration verification (CCV) standard.
2	Evaluate the system performance check compounds (SPCCs) : Each SPCC in the calibration verification (CCV) standard must meet the minimum response factor criteria for 8270C or 8270D in the initial calibration.
3	Evaluate the minimum response factors of each of the most common target analytes in the calibration verification standard (same as SPCCs).
4	Evaluate the calibration check compounds (CCCs) for method criteria. For 8270D or for shortened compound lists, all target analytes must meet $\pm 20\%$ criteria. Use the initial calibration criteria.
5	Evaluate the internal standard retention times in the CCV.
6	Evaluate the internal standard responses .
7	Analyze an extraction blank after the continuing calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants.

10.3 Sample Analysis: Refer to Acceptable Manual Integration Practices / CA-Q-S-002.

1	Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 μ L [RVE/LVI: 2 μ L] of the internal standard solution to the 0.5 mL concentrated sample extract.														
2	Inject a 1 μ L [RVE/LVI: 5 μ L] aliquot of the sample extract into the GC/MS system. The volume to be injected contains 50 ng of base/neutral and 50 ng of acid surrogates (assuming 100% recovery).														
3	The recommended sequence for a 20-sample batch is as follows: <table border="1"> <tr> <td>1</td><td>DFTPP Tuning Criteria /DDT Breakdown/Tailing Factors*</td></tr> <tr> <td>2</td><td>CCV</td></tr> <tr> <td>3</td><td>Method Blank</td></tr> <tr> <td>4</td><td>LCS</td></tr> <tr> <td>5</td><td>Matrix Spike</td></tr> <tr> <td>6</td><td>Matrix Spike</td></tr> <tr> <td>7</td><td>Samples 1-20</td></tr> </table>	1	DFTPP Tuning Criteria /DDT Breakdown/Tailing Factors*	2	CCV	3	Method Blank	4	LCS	5	Matrix Spike	6	Matrix Spike	7	Samples 1-20
1	DFTPP Tuning Criteria /DDT Breakdown/Tailing Factors*														
2	CCV														
3	Method Blank														
4	LCS														
5	Matrix Spike														
6	Matrix Spike														
7	Samples 1-20														

*Not used for SIM.

4	If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed in the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (40 µg/mL, unless a more sensitive GC/MS system is being used, e. g., 2 µg/mL for SIM).
5	Evaluate the specific internal standard response. Dilutions may be required to meet this criterion. Notes: Specific analytes associated with an internal standard within -50 to +100% from the last calibration verification (CCV) may be reported with approval from the supervisor or manager even if other internal standards in that analysis are outside limits. Only analytes associated with the internal standard(s) within limits may be reported from that analysis.
6	The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. Multiple ions are used for compound identification; see Attachment 2. Secondary ions may drop below 30% relative intensity at concentrations less than 1 µg/mL.

10.5 Qualitative analysis

- The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be kept up to date and obtained through analysis of known standards on the instrument using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Attachments 1 and 2 list the primary and secondary ions for each analyte. Compounds are identified when the following criteria are met.
- The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time is accepted as meeting this criterion.
- The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. **Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.** When two or more analytes that co-elute share secondary ions, and all the characteristic secondary ions for the target analyte are present but outside the $\pm 30\%$ relative intensity, the compound is reported as positive if there is no interference with the primary quantitation ion. If co-eluting peaks share the primary ion, the analyte may only be reported as a co-eluting pair. (See Attachment 1.)
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i. e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analyses co-elute (i. e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound. The analyst must

carefully weigh the background spectrum and the spectrum of any co-eluting analytes whenever assessing a potential hit. Analyst experience in interpreting mass spectral data and the above specified guidelines are used together to interpret difficult matrices. If all of the ions associate with the reference spectrum for the target analyte are present and within the $\pm 30\%$ criteria, a positive result is assumed even in the presence of extraneous ion fragments without presumptive evidence (all ions associated with the target analyte are also present in the interfering peak) for a negative identification.

- Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights for 8270C and 50% of the average of the two peak heights for 8270D samples. Mathematically, the two equations used are equivalent. Verification is performed on a mid-level control each day of use. Otherwise, structural isomers are identified as isomeric pairs. (See Attachment 1.)
- For samples containing components not associated with the calibration standards or the requested target list, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines do not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.
 - For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:
 - 1) Relative intensities of major ions in the reference spectrum (ions $>10\%$ of the most abundant ion) are present in the sample spectrum.
 - 2) The relative intensities of the major ions agree within $\pm 20\%$. **Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.**
 - 3) Molecular ions present in the reference spectrum are present in the sample spectrum.
 - 4) Ions present in the sample spectrum but not in the reference spectrum are reviewed for possible background contamination or presence of co-eluting compounds.
 - 5) Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6 Quantitative analysis

- Once a compound has been identified, the quantitation of that compound is based on the integrated abundance of the primary characteristic ion from the EICP.
- If the RSD of a compound's response factor is 15% for 8270C and 20% for 8270D, or less, then the concentration in the extract is determined using the average response factor (RF) from initial calibration data. If greater than the criteria, use linear regression.
- Where applicable, the concentration of any non-target compounds identified in the sample is estimated. The same formulae are used with the following modifications: The areas A_x and A_t are from the total ion chromatograms, and the RF for the compound is assumed to be 1.
- The resulting concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.
-

10.7 Instrument Maintenance

Careful examination of the standard chromatogram indicates whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. Re-calibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. In addition, significant maintenance activities or hardware changes may also require re-calibration. These significant maintenance activities include, changing, replacing, or reversing the column; cleaning the MS source; changing the electron multiplier; or injector port.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\% \text{ Recovery} = \frac{\text{Measured concentration} \times 100}{\text{Known concentration}}$$

11.2 Precision (RPD)

$$\text{RPD} = \frac{\text{Absolute value (orig. sample value - dup. sample value)} \times 100}{(\text{Orig. sample value} + \text{dup. sample value})/2}$$

11.3 Breakdown Calculation:

$$\% \text{ Breakdown of DDT} = \frac{\text{Sum of degradation peak areas (DDD + DDE)} \times 100}{\text{Sum of all peak areas (DDT + DDE + DDD)}}$$

1.4 Response Factor

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Concentration of the analyte or surrogate, in $\mu\text{g/L}$.

C_{is} = Concentration of the internal standard, in $\mu\text{g/L}$.

11.5 Mean Response Factor, Standard Deviation, Relative Standard Deviation

$$RF_{\text{mean}} = \frac{\sum_{i=1}^n RF_i}{n}$$

$$SD = \frac{\sum_{i=1}^n (RF_i - RF_{\text{mean}})^2}{n-1}$$

$$RSD = \frac{SD \times 100}{RF_{\text{mean}}}$$

11.6 % Difference, % Drift

$$\% \text{ Difference} = \frac{(RF_v) - (\text{Avg. RF}) \times 100}{(\text{Avg. RF})}$$

RF_v = RF from verification standard

Avg. RF = Average RF from Initial Calibration.

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value} \times 100}{\text{True Value}}$$

11.7 Linear Calibration Using a Least Squares Regression: This approach is not used for analytes that meet the RSD limits. For calibration, x is the mass of the analyte in the sample aliquot introduced into the instrument and y is the area or the response, as in:

$$x = C_s \text{ and } y = A_s$$

A linear least squares regression attempts to construct a linear equation of the form:

$$y = ax + b$$

by minimizing the differences between the observed results (y_i , the instrument response) and the predicted results (y_i' , the response calculated from the constructed equation). The regression equation is:

$$y_i' = ax_i + b$$

a = regression coefficient or the slope of the line.

b = the y-intercept.

y_i' = predicted (or calculated) response for the i^{th} calibration standard.

x_i = mass of analyte in the i^{th} calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^n (y_i - y_i')^2$$

n = total number of calibration points. The regression calculations attempt to minimize this sum of the squares, hence the name "least squares regression."

Weighting the sum of the square of the differences may significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^n w_i (y_i - y_i')^2$$

w_i = weighting factor for the i^{th} calibration standard ($w=1$ for unweighted least squares regression).

y_i – observed instrument response (area) for the i^{th} calibration standard.

y_i' = predicted (or calculated) response for the i^{th} calibration standard.

n = total number of calibration standards.

The mathematics used in least squares regression has a tendency to favor numbers of larger value over numbers of smaller value. Thus the regression curves that are generated tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a weighting factor which reduces this tendency can be used. Examples of allowed weighting factors which can place more emphasis on numbers of smaller value are:

$$w_i = 1/x_i \quad \text{or} \quad w_i = 1/x_i^2$$

Do not include the origin (0, 0) as an extra calibration point. Reprocess each calibration standard as an unknown to determine the best fit model. Each calibration point above the RL must be $\pm 15\%$ true (8000B) or $\pm 20\%$ true (8000C); the RL-level standard must be $\pm 30\%$ true.

The regression calculation generates a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995 or $r^2 \geq 0.990$.

11.8 Coefficient of Determination

$$r^2 = \frac{(\sum xy)^2}{\sum x^2 \sum y^2}$$

Correlation Coefficient

$$r = \frac{(\sum xy)}{\sqrt{\sum x^2 \sum y^2}}$$

y = Response or Response ratio

x = Concentration

11.9 Calculation

- For **aqueous** samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{A_x V_t D}{\text{RF}_{\text{mean}} V_s} \quad \text{or} \quad \frac{(\mu\text{g/mL from instrument}) (D)(1000)}{\text{mL extracted}}$$

A_x = Area of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (mL).

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, $D = 1$. The dilution factor is always dimensionless.

RF_{mean} = Mean response factor from the initial calibration (area/concentration).

V_s = Volume of the aqueous sample extracted in mL.

- For **non-aqueous** samples:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{A_x V_t D}{\text{RF}_{\text{mean}} W_s} \quad \text{or} \quad \frac{(\mu\text{g/mL from instrument}) (D)(1000)}{\text{g extracted}}$$

A_x , V_t , D , RF_{mean} are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL): The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in SOP Determination of Method Detection Limits / NV08-202. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capability: The laboratory demonstrates initial proficiency by generating data of acceptable accuracy and precision for target analyses in a clean matrix. The laboratory also repeats the operation whenever new staff is trained or significant changes in instrumentation are made and on an annual basis thereafter. See the training section of TestAmerica-Nashville's QA Manual and SOP Training / NV08-199 for information on how to accomplish this demonstration.

12.3 Training Requirements: Demonstration of Capability is performed initially when learning the method and annually thereafter. Four Laboratory Control Samples resulting in an average % recovery within the control limits and a precision less than the quality control maximum are required.

12.4 Proficiency Testing Studies: The laboratory participates in formal proficiency testing (PT) studies, where solutions of unknown concentrations are analyzed and the performance of all participants is compared. See the QA department for the results of recent PT studies.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i. e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be stored, managed, and disposed of in accordance with all federal and state laws and regulations. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the QA Manual and SOP Waste Disposal / NV10-83.

14.2 Wastestreams Produced by the Method:

- Dispose of waste extracts in the waste solvent drum.

15.0 References / Cross References

15.1 Method 8270C, SW-846 Update III Revision 3, December 1996 and **Method 8270D**, Update IV, Revision 4, February 2007.

15.2 Method 8000B, SW-846, Revision 2, December 1996, **Method 8000C**, Revision 3, March 2003.

15.3 TestAmerica Nashville's Quality Assurance Manual.

15.4 Corporate Environmental Health and Safety Manual (CW-E-M-001).

15.5 SOPs: Acceptable Manual Integration Practices / CA-Q-S-002, Selection of Calibration Points / CA-T-P-002, Calibration Curves (General) / CA-Q-S-005, Waste Disposal / NV10-83, Training Procedures for Environmental Technical Staff / NV08-199, Determination of Method

Detection Limits / NV08-202, Reagent and Standard Purchase / NV08-214, 3550 / NV03-23, and 3510 / NV03-24, 3541 / NV03-231, 3580 / NV03-106, 8270/NVOH04-22.

15.6 Controlled Document: QAF-45, TestAmerica Nashville – Acronyms, Keywords, and Definitions.

15.7 Corporate Quality Memorandum No. CA-Q-QM-005, May 19, 2010.

16.0 Method Modifications

Item	Modification
1	See Attachment 5 for the State of Ohio specific criteria.
2	See Attachment 6 for the State of Missouri DRO, CA LUFT DRO by GC/MS.
3	Verify with state certifications the correct version of this method to report. Analyze and report by 8270D for Canadian, NJ, NC, OK, SC, and WV samples.
4	SIM is not allowed for South Carolina samples unless pre-approved by the state on a project-specific basis.

17.0 Attachments

Attachment 1, Characteristic Ions for Semivolatile Compounds^a

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
1,4-Dioxane	2.568	88	58
n-Nitrosodimethylamine	2.700	74	42, 44
Pyridine	2.714	79	52
2-Picoline	3.464	93	66, 92
n-Nitrosomethylethylamine	3.558	88	42, 43, 56
2-Fluorophenol (surr)	3.687	112	64
Methyl methanesulfonate	3.764	80	79, 65, 95
n-Nitrosodiethylamine	4.009	102	42, 57, 44, 56
Ethyl methanesulfonate	4.197	79	109, 97, 45, 65
Hexachloropropene	4.261	213	211, 215, 117, 106, 141
Phenol-d ₅ (surr)	4.266	99	42, 71
Aniline	4.270	93	66, 65
Bis(2-chloroethyl) ether	4.294	93	63, 95
Phenol	4.275	94	65, 66
2-Chlorophenol	4.345	128	64, 130
1,3-Dichlorobenzene	4.425	146	148, 113
1,4-Dichlorobenzene-d ₂ (IS)	4.444	152	150, 115
1,4-Dichlorobenzene	4.454	146	148, 113
Pentachloroethane	4.474	117	165, 167, 119
Benzyl alcohol	4.543	79	108, 77
n-Decane	4.550	57	
1,2-Dichlorobenzene	4.571	146	148, 113
2-Methylphenol	4.628	108	107, 77, 79, 90
Bis(2-chloroisopropyl) ether	4.632	45	77, 79
N-Nitrosodi-n-propylamine	4.717	130	42, 101, 70
3, 4-Methylphenol	4.717	107	108, 77, 79, 90
Hexachloroethane	4.764	117	201, 199
Nitrobenzene-d ₅ (surr)	4.806	82	128, 54
Nitrobenzene	4.816	77	123, 65
n-Nitrosopyrrolidine	4.907	102	41, 42, 68, 69
Acetophenone	4.912	105	71, 51, 120
n-Nitrosomorpholine	4.916	108	116, 86
o-Toluidine	4.940	106	107, 77, 51, 79

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Isophorone	4.957	82	95, 138
2-Nitrophenol	5.018	139	109, 65
2,4-Dimethylphenol	5.037	122	107, 121
Bis(2-chloroethoxy)methane	5.088	93	95, 123
n-Nitrosopiperidine	5.114	114	42, 55, 56, 41
Benzoic acid	5.116	105	122, 77
2,4-Dichlorophenol	5.168	162	164, 98
1,2,4-Trichlorobenzene	5.215	180	182, 145
Naphthalene-d ₈ (IS)	5.248	136	68
Naphthalene	5.257	128	129, 127
o,o,o-Triethylphosphorthioate	5.302	198	80, 53, 54, 164, 63
4-Chloroaniline	5.304	127	129, 65, 92
Hexachlorobutadiene	5.370	225	223, 227
a,a-Dimethylphenethylamine	5.372	98	91, 65, 134, 42
2,6-Dichlorophenol	5.523	162	
Hexachloropropene	5.556	213	211, 215, 117, 106, 141
4-Chloro-3-methylphenol	5.615	142	107, 144
2-Methylnaphthalene	5.704	142	141
n-Nitrosodi-n-butylamine	5.729	84	57, 41, 116, 158
1,4-Phenylenediamine	5.734	198	80, 53, 54, 52
1-Methylnaphthalene	5.779	142	141, 115
Hexachlorocyclopentadiene	5.854	237	235, 272
Isosafrole (trans)	5.861	162	131, 104, 77
2,4,6-Trichlorophenol	5.911	196	198, 200
2,4,5-Trichlorophenol	5.944	196	198, 97, 132, 99
2-Fluorobiphenyl (surr)	5.953	172	171
2-Chloronaphthalene	6.063	162	127, 164
Isosafrole (cis)	6.054	162	131, 104, 77
1,2,4,5-Tetrachlorobenzene	6.063	216	214, 179, 108, 143, 218
2-Nitroaniline	6.118	138	92, 65
2,3-Dichloroaniline	6.134	161	90, 63
Safrole	6.204	162	104, 77, 103, 135
Dimethyl phthalate	6.245	163	194, 164
1-Chloronaphthalene	6.284	162	127, 164
2,6-Dinitrotoluene	6.296	165	63, 89, 121
Acenaphthylene	6.320	152	151, 153
1,4-Naphthoquinone	6.374	158	104, 102, 76, 50, 130
3-Nitroaniline	6.404	138	108, 92
Acenaphthene	6.447	154	153, 152
2,4-Dinitrophenol	6.470	184	63, 154
1,3-Dinitrobenzene	6.486	168	76, 50, 75, 92, 122
4-Nitrophenol	6.527	65	109, 139
Dibenzofuran	6.560	168	139
2,4-Dinitrotoluene	6.574	165	63, 89, 182
Acenaphthene-d ₁₀ (IS)	6.656	164	162, 160
Diethyl phthalate	6.738	149	177, 150
4-Chlorophenyl phenyl ether	6.790	204	206, 141
Fluorene	6.799	166	165, 167
Pentachlorobenzene	6.806	250	252, 108, 248, 215, 254
4-Nitroaniline	6.837	138	65, 108, 92, 80, 39
1-Naphthylamine	6.844	143	115, 89, 63

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
4,6-Dinitro-2-methylphenol	6.865	198	51, 105, 182, 77
n-Nitrosodiphenylamine	6.879	169	168, 167
2-Naphthylamine	6.895	143	115, 116
2,3,4,6-Tetrachlorophenol	6.900	232	131,230,166,234,168
1,2-Diphenylhydrazine	6.903	77	105, 182
2,4,6-Tribromophenol (surr)	6.987	330	332, 141
Thionazine	7.027	107	96, 97, 143, 79, 68
5-Nitro-o-toluidine	7.051	152	77, 79, 106, 94
Diphenylamine	7.107	168	169, 167
4-Bromophenyl phenyl ether	7.138	248	250, 141
Hexachlorobenzene	7.255	284	142, 249
Sulfotepp	7.276	322	97, 202
1,3,5-Trinitrobenzene	7.314	213	74, 120, 91, 63
Diallate (trans)	7.337	86	234, 43, 70
Phenacetin	7.337	108	180,179,109,137,80
Phorate	7.347	75	121, 97, 93, 260
Pentachlorophenol	7.387	266	264, 268
Diallate (cis)	7.403	86	234, 43, 70
Dimethoate	7.474	87	93, 125, 143, 229
Phenanthrene-d ₁₀ (IS)	7.476	188	94, 80
Phenanthrene	7.495	178	179, 176
Anthracene	7.528	178	176, 179
4-Aminobiphenyl	7.568	169	168, 170, 115
n-Octadecane	7.586	58	71, 85
Pronamide	7.619	173	175, 145, 109, 147
Carbazole	7.641	167	139, 84
Pentachloronitrobenzene	7.676	237	142,214,249,295,265
Disulfoton	7.723	88	97, 89, 142, 186
Dinoseb	7.737	211	163, 147, 117, 240
Di-n-butyl phthalate	7.914	149	150, 104
Methyl parathion	8.000	109	125, 263, 79, 93
Parathion	8.292	109	97, 291, 139, 155
4-Nitroquinoline-1-oxide	8.310	190	101, 128, 75, 116
Methapyrilene	8.371	58	50, 191, 71
Fluoranthene	8.374	202	100, 101, 203
Benzidine	8.464	184	92, 185
Isodrin	8.522	193	66, 195, 263,265,147
Pyrene	8.543	202	100, 101, 200, 203
Terphenyl-d ₄ (surr)	8.652	244	122, 212
Aramite	8.870	191	319, 334, 197, 321
Dimethylaminoazobenzene	9.001	120	77, 105, 148, 42
Butyl benzyl phthalate	9.028	149	91, 206
Chlorobenzilate	9.034	139	253, 111, 141
Hexachlorophene	9.070	185	209,406
3,3'-Dimethylbenzidine	9.251	212	106, 196, 180
Bis (2-ethylhexyl) adipate	9.298	129	57, 112, 147
4,4'-Methylenebis (2-chloroaniline)	9.301	231	266, 140, 77
Kepone	9.316	272	274,237,178,143,270
3,3'-Dichlorobenzidine	9.423	252	254, 126
Benz(a)anthracene	9.446	228	229, 226
2-Acetylaminofluorene	9.453	181	180, 223, 152

Compound	Retention Time (minutes)	Primary Ion	Secondary Ion(s)
Chrysene-d ₁₂ (IS)	9.456	240	120, 36
Chrysene	9.474	228	226, 229
Bis(2-ethylhexyl) phthalate	9.474	149	167, 279
Di-n-octyl phthalate	9.926	149	167, 43
Benzo(b)fluoranthene	10.292	252	253, 125
3-Methylcholanthrene	11.305	268	252, 253, 126, 134, 113
Benzo(k)fluoranthene	10.311	252	253, 125
Benzo(a)pyrene	10.579	252	253, 125
7,12-Dimethylbenz(a)anthracene	10.600	256	241, 239, 120
Perylene-d ₁₂ (IS)	10.631	264	260, 265
Indeno(1,2,3-c,d)pyrene	11.778	276	138, 277
Dibenz(a,h)anthracene	11.783	278	139, 279
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Dibenz(a,j)acridine	11.987	279	280, 277, 250
Benzo(g,h,i)perylene	12.107	276	138, 277
IS = internal standard			
surr = surrogate			
^a See Attachment 2 for Retention Times and Ions used with SIM			

Attachment 2, Characteristic Ions for PAH Compounds Using SIM

Compounds	RT	Primary	Secondary*
1,4-Dichlorobenzene-d ₄	6.66	152	
2-Fluorophenol	5.471	112	64
Phenol-d ₅	6.251	99	71.1
Naphthalene-d ₈	8.24	136	
Nitrobenzene-d ₅	7.32	82.1	128.1
Naphthalene	8.27	128.1	129.1
2-Methylnaphthalene	9.13	142.1	141.1
1-Methylnaphthalene	9.42	142.1	141.1
Acenaphthene-d ₁₀	10.91	164.1	
2-Fluorobiphenyl	9.82	172.1	
Acenaphthylene	10.67	153	151.1
Acenaphthene	10.958	15.1	154.1
Fluorene	11.7	166.1	167.1
Phenanthrene-d ₁₀	13.3	188	
2,4,6-Trichlorophenol	12.167	329.8	331.8
Phenanthrene	13.33	178.2	176.2
Anthracene	13.4	178.2	176.2
Fluoranthene	15.28	202.2	101.1
Chrysene-d ₁₂	17.64	240.1	
Pyrene	15.66	202.2	101.1
Terphenyl-d ₁₄	15.89	244.2	
Benzo(a)anthracene	17.61	228.2	229.2
Chrysene	17.68	228.2	229.2
Perylene-d ₁₂	20.2	264.2	
Benzo(b)fluoranthene	19.45	252.2	126.1
Benzo(k)fluoranthene	19.49	252.2	126.1
Benzo(a)pyrene	20.08	252.2	126.1
Indeno(1,2,3-cd)pyrene	22.69	276.2	277.2
Dibenzo(ah)anthracene	22.7	278.2	279.2

Compounds	RT	Primary	Secondary*
Benzo(ghi)perylene	23.43	276.2	277.2
Internal standards are in bold .			

Attachment 3, SIM Mass Groups

Mass Group	Compound	RT	Primary	Secondary	Dwell Time
1	2-Fluorophenol	5.42	112	64	25 ms
	Phenol-d ₅	6.2	99	71.1	
	1,4-Dichlorobenzene-d ₄	6.6	152		
	Nitrobenzene-d ₅	7.26	82.1	128.1	
4.20 min.					
2	Naphthalene-d ₈	8.17	136		50 ms
	Naphthalene	8.2	128.1	129.1	
	2-Methylnaphthalene	9.19	142.1	141.1	
	1-Methylnaphthalene	9.35	142.1	141.1	
	2-Fluorobiphenyl	9.75	172.1		
5.35 min.					
3	Acenaphthylene	10.6	152.1	151.1	25 ms
	Acenaphthalene-d ₁₀	10.83	164.1		
	Acenaphthene	10.88	153.1	154.1	
	Fluorene	11.69	166.1	167.1	
	2,4,6-Tribromophenol	12.11	329.8	331.8	
6.55 min.					
4	Phenanthrene-d ₁₀	13.21	188		50 ms
	Phenanthrene	13.25	178.2	176.2	
	Anthracene	13.32	178.2	176.2	
	Fluoranthene	15.21	202.2	101.1	
	Pyrene	15.58	202.2	101.1	
	Terphenyl-d ₁₄	15.81	244.2		
7.75 min.					
5	Benzo(a)anthracene	17.52	228.2	229.2	100 ms
	Chrysene-d ₁₂	17.55	240.1		
	Chrysene	17.59	228.2	229.2	
9.85 min.					
6	Benzo(b)fluoranthene	19.34	252.2	126.1	100 ms
	Benzo(k)fluoranthene	19.38	252.2	126.1	
	Benzo(a)pyrene	19.96	252.2	126.1	
	Perylene-d ₁₂	20.07	264.2		
10.65 min.					
7	Indeno(1,2,3-cd)pyrene	22.5	276.2	277.2	100 ms
	Dibenzo(a,h)anthracene	22.52	278.2	279.2	
	Benzo(g,h,i)perylene	23.21	276.2	277.2	
12.20 min.					

Attachment 4, 8270D Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Attachments 1 and 2.

Compound	Minimum RF	Compound	Minimum RF
Benzaldehyde	0.010	4-Nitrophenol	0.010
Phenol	0.800	Dibenzofuran	0.800

Compound	Minimum RF	Compound	Minimum RF
Bis(2-chloroethyl)ether	0.700	2,4-Dinitrotoluene	0.200
2-Chlorophenol	0.800	Diethyl phthalate	0.010
2-Methylphenol	0.700	1,2,4,5-tetrachlorobenzene	0.010
2,2'-Oxybis-(1-chloropropane)	0.010	4-Chlorophenyl-phenyl ether	0.400
Acetophenone	0.010	Fluorene	0.900
4-Methylphenol	0.600	4-Nitroaniline	0.010
n-Nitroso-di-n-propylamine	0.500	4,6-Dinitro-2-methylphenol	0.010
Hexachloroethane	0.300	4-Bromophenyl-phenyl ether	0.100
Nitrobenzene	0.200	n-Nitrosodiphenylamine	0.010
Isophorone	0.400	Hexachlorobenzene	0.100
2-Nitrophenol	0.100	Atrazine	0.010
2, 4-Dimethylphenol	0.200	Pentachlorophenol	0.050
Bis(2-chloroethoxy)methane	0.300	Phenanthrene	0.700
2,4-Dichlorophenol	0.200	Anthracene	0.700
Naphthalene	0.700	Carbazole	0.010
4-Chloroaniline	0.010	Di-n-butyl phthalate	0.010
Hexachlorobutadiene	0.010	Fluoranthene	0.600
Caprolactam	0.010	Pyrene	0.600
4-Chloro-3-methylphenol	0.200	Butyl benzyl phthalate	0.010
2-Methylnaphthalene	0.400	3,3'-Dichlorobenzidine	0.010
Hexachlorocyclopentadiene	0.050	Benzo(a)anthracene	0.800
2,4,6-Trichlorophenol	0.200	Chrysene	0.700
2,4,5-Trichlorophenol	0.200	Bis-(2-ethylhexyl)phthalate	0.010
1,1'-Biphenyl	0.010	Di-n-octyl phthalate	0.010
2-Chloronaphthalene	0.800	Benzo(b)fluoranthene	0.700
2-Nitroaniline	0.010	Benzo(k)fluoranthene	0.700
Dimethyl phthalate	0.010	Benzo(a)pyrene	0.700
2,6-Dinitrotoluene	0.200	Indeno(1,2,3-cd)pyrene	0.500
Acenaphthylene	0.900	Dibenz(a,h)anthracene	0.400
3-Nitroaniline	0.010	Benzo(g,h,i)perylene	0.500
Acenaphthene	0.900	2,3,4,6-Tetrachlorophenol	0.010
2,4-Dinitrophenol	0.010		

Attachment 5, State of Ohio Specific Criteria.

Only those compounds in the original EPA Method 8270C may be reported. Any compounds in this SOP in italics in Section 1 are not part of the original 8270C method. Run Ohio VAP samples according to SOP 8270/NVOH04-22.

Attachment 6, Missouri Department of Natural Resources (and CA LUFT) require(s) that DRO be analyzed by GC/MS.

- Tuning and frequency requirements are the same as in 8270, omitting DDT, Pentachlorophenol, and Benzidine.
- Extract water samples per SOP 3510 / SA03-24 and solid samples per SOP 3550 / SA03-23.
- Only base/neutral surrogates are needed.
- GC/MS mass range is 35-550 m/z .
- Use a five-point calibration curve with 1:1 unleaded gasoline and #2 diesel fuel at 1,000 $\mu g/mL$ each in Methylene chloride.

- Retention time windows set using C₁₀, C₂₁, and C₃₅. For DRO, set RT 0.1 minutes after C₁₀ to 0.1 minutes after C₂₁. For ORO, set RT 0.1 minutes after C₂₁ to 0.1 minutes after C₃₅. Verify RT daily (24 hours) by running component standard.
- Quantitative using baseline-to-baseline, not valley-to-valley. The Total Ion Chromatogram must be used to quantitate.
- The Response Factor determined for DRO (C₁₀-C₂₁) must be used for C₂₁-C₃₅.
- Subtract area from any Internal Standard and surrogates.
- % RSD ≤ 20.
- Run a CCV at the beginning and end of each batch; it must contain all targets reported, at mid-point of calibration, % D ≤ 20.
- Run a Method Blank every extraction batch, and LCS and MS/MSD per batch.
- May reprocess file to quantitate PAH if needed. For individual targets, % RSD ≤ 15.
- Quantitation of DRO must be by external standard.

18.0 Revision History

- Revision 12, 22 October 2008
 - Integration for TestAmerica and STL operations.
 - Insert corrective action procedures
 - To incorporate Update IV criteria.
- Revision 13, 9 October 2009
 - Consolidation of text, general editing.
 - Add Appendix IX and miscellaneous compound details.
 - Distinguish 8270C versus 8270D.
- Revision 14, 30 September 2011
 - Organizational changes.
 - Add amendments 13a and 13b.
 - Add reference to SOP 3541 for concrete and SOP Calibration Curves (General).
 - Add QAF-45 and Section 14.2.
 - Remove WY as a state requiring QC every 10 samples.
 - Change Attachment 5 to refer analysts to OH8270 SOP.
 - Add Attachment 7
 - Add option to run LLCCV to show sensitivity.
 - Add note about low-level calibration standard for SIM WI samples.
 - Lower several report limits.
 - Specify GC resolution between two isomer peaks for 8270C versus 8270D.
- Revision 15, 31 December 2012
 - Organizational changes.
 - Incorporation of amendments 14a, b, c.
 - OK no longer limits batch size to 10 samples.
 - Specify that $r^2 \geq 0.990$.
 - Substitute LIMS for the Control Limits Manual.
 - Distinguish between the RSD maximum for 8270C and 8270D. For 8270D, all targets are treated as CCCs.
 - Add re-fitting text to the linear calibration section.
 - Add Reduced Volume Extraction / Large Volume Injection (RVE / LVI).

ATTACHMENT C3

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

Title: Integrys Multi-Site QAPP**Revision Number: 2****Addendum Date: 7/23/14****Page 1 of 3****Reference Limits and Evaluation Table**

Laboratory: Test America

Matrix: Water

Analytical Group/Method: Semivolatile Organic Compounds - 8270C SIM High Volume Injection

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit ² (applicable units)	Achievable Laboratory Limits ¹	
				MDLs	QLs
4-Nitroaniline	100-01-6	3.4			
4-Nitrophenol	100-02-7	no value			
Benzaldehyde	100-52-7	3700			
4-Bromophenylphenyl ether	101-55-3	no value			
Caprolactam	105-60-2	18000			
2,4-Dimethylphenol	105-67-9	100			
3&4-Methylphenol (m&p)	106-44-5	67			
4-Methylphenol	106-44-58	25			
4-Chloroaniline	106-47-8	28			
2,2'-Oxybis(1-chloropropane)	108-60-1	0.32			
Phenol	108-95-2	100			
bis(2-Chloroethyl) ether	111-44-4	10			
bis(2-Chloroethoxy)methane	111-91-1	110			
bis(2-Ethylhexyl)phthalate	117-81-7	6			
Di-n-octylphthalate	117-84-0	140			
Hexachlorobenzene	118-74-1	0.06			
Anthracene	120-12-7	0.059			
2,4-Dichlorophenol	120-83-2	21			
2,4-Dinitrotoluene	121-14-2	0.02			

Pyrene	129-00-0	0.29			
Dimethylphthalate	131-11-3	no value			
Dibenzofuran	132-64-9	37			
Atrazine	1912-24-9	3			
Benzo(g,h,i)perylene	191-24-2	0.013			
Indeno(1,2,3-cd)pyrene	193-39-5	0.008			
Benzo(b)fluoranthene	205-99-2	0.019			
Fluoranthene	206-44-0	0.2			
Benzo(k)fluoranthene	207-08-9	0.018			
Acenaphthylene	208-96-8	8.77			
Chrysene	218-01-9	0.058			
Benzo(a)pyrene	50-32-8	0.027			
2,4-Dinitrophenol	51-28-5	14			
4,6-Dinitro-2-methylphenol	534-52-1	2.9			
Dibenz(a,h)anthracene	53-70-3	0.008			
Benzo(a)anthracene	56-55-3	0.064			
2,3,4,6-Tetrachlorophenol	58-90-2	1100			
4-Chloro-3-methylphenol	59-50-7	3700			
2,6-Dinitrotoluene	606-20-2	0.31			
N-Nitroso-di-n-propylamine	621-64-7	1.8			
Hexachloroethane	67-72-1	7			
4-Chlorophenylphenyl ether	7005-72-3	no value			
Hexachlorocyclopentadiene	77-47-4	50			
Isophorone	78-59-1	1400			
Acenaphthene	83-32-9	1.6			
Diethylphthalate	84-66-2	5600			
Di-n-butylphthalate	84-74-2	700			
Phenanthrene	85-01-8	0.55			
Butylbenzylphthalate	85-68-7	1400			
N-Nitrosodiphenylamine	86-30-6	3.2			
Fluorene	86-73-7	1.12			
Carbazole	86-74-8	no value			
Hexachloro-1,3-butadiene	87-68-3	0.86			
Pentachlorophenol	87-86-5	1			
2,4,6-Trichlorophenol	88-06-2	10			
2-Nitroaniline	88-74-4	370			
2-Nitrophenol	88-75-5	no value			
Naphthalene (cancer)	91-20-3	0.6			

2-Methylnaphthalene	91-57-6	150			
2-Chloronaphthalene	91-58-7	2900			
3,3'-Dichlorobenzidine	91-94-1	20			
Biphenyl (Diphenyl)	92-52-4	1800			
2-Methylphenol (o-Cresol)	95-48-7	350			
2-Chlorophenol	95-57-8	35			
1,2,4,5-Tetrachlorobenzene	95-94-3	11			
2,4,5-Trichlorophenol	95-95-4	700			
Acetophenone	98-86-2	3700			
Nitrobenzene	98-95-3	3.5			
3-Nitroaniline	99-09-2	2.1			
Perylene	NA	0.026			

¹Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. Limits vary over time per lab performance tests and capabilities. As Multi-Site Screening Levels/Project Quantitation Limits are updated, lab's ability to achieve new limits are verified before work proceeds.

²Project Quantitation Limits/Multi-Site Screening Levels are updated approximately every six months per the USEPA-approved Multi-Site Risk Assessment Framework for the Integrys Multi-Site Manufactured Gas Plant Program. As Multi-Site Screening Levels/Project Quantitation Limits are updated, lab's ability to achieve new limits are verified before work proceeds.

ATTACHMENT C4

**QAPP Worksheet #24
(UFP-QAPP Manual Section 3.2.2)**

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference¹
GC/MS	Tune (BFB, DFTPP)	Prior to initial calibration or continuing calibration; every 12 hours	Refer to SOP	Correct problem; re-analyze tune	Analyst	
GC/MS	Initial Calibration	Prior to sample analysis or as needed	VOC : CCC \leq 30% RSD; SPCC \geq 0.300 or 0.100; all other targets <15% RSD; linear $r \geq$ 0.995 SVOC: CCC \leq 30% RSD; SPCC \geq 0.05; all other targets grand mean <15% RSD; linear $r \geq$ 0.995	Correct problem; repeat initial calibration	Analyst	
GC/MS	Continuing Calibration	Daily, before sample analysis and every 12 hours of tune time	VOC : CCC \leq 20% DIFF/Drift SPCC \geq 0.300 or 0.100; SVOC: CCC \leq 20% DIFF/Drift SPCC \geq 0.05	Correct problem and repeat CCV and associated samples; repeat initial calibration if necessary and CCV and samples; may report non-detects if biased high.	Analyst	
Metals (ICP)	Initial Calibration	Daily initial calibration prior to sample analysis	$r \geq$ 0.995	Correct problem; repeat initial calibration	Analyst	
Metals (ICP)	Continuing Calibration	After every 10 readings and end of the analytical sequence	All analytes within 10% of expected value	Correct problem and re-analyze affected elements and bracketed samples; may report non-detects if biased high	Analyst	

¹ Lab Notes: See Quality System Manual and specific Analytical Standard Operating Procedures (SOPs) for full details

**QAPP Worksheet #25
(UFP-QAPP Manual Section 3.2.3)**

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference¹
Hewlett Packard GC/MS	Routine	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	Check GC / Entech Column Interface Check Nitrogen Tank Volume Check Nitrogen Valves Software and Valves Cut 2-3 inches from GC Column	As required	Passing calibration	Perform maintenance, check standards, recalibrate	Laboratory Analyst	
Thermo Scientific ICP	Routine	Inspect sample introduction system, nebulizer, torch and injection tubing	Check for salt build up, dirt and debris that may restrict flow	As required	Passing calibration	Perform maintenance, replace/clean tubing, check standards, recalibrate	Laboratory Analyst	

¹Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

Lab Note: All maintenance procedures will follow the Laboratory Quality System Manual and Analytical Standard Operating Procedures (SOP)



Enclosure E
Appendix C of the
Manitowoc Step II Site-
Specific Work Plan,
Revision 2 (NRT, April 30,
2012)

**APPENDIX C of Manitowoc Step II
Site-Specific Work Plan**

**POREWATER PAH TARGET ANALYTES, REPORTING
LIMITS, AND STANDARD OPERATING PROCEDURE
FOR PAH ANALYSIS BY SOLID PHASE
MICROEXTRACTION (SPME)**

META ENVIRONMENTAL, INC.

Table 1. META Environmental, Inc.**Compound Lists and Reporting Limits for PAHs and Alkylated PAHs in Sediment by GC/MS/SIM and Porewater by SPME/GC/MS/SIM**

34 PAHs	Sediments (µg/kg)	Waters (µg/L)
Naphthalene	5	0.8
C1-Naphthalenes ¹	5	0.4
C2-Naphthalenes	5	0.6
C3-Naphthalenes	5	0.9
C4-Naphthalenes	5	1.1
Acenaphthylene	5	0.2
Acenaphthene	5	0.2
Fluorene	5	0.12
C1-Fluorenes	5	0.16
C2-Fluorenes	5	0.2
C3-Fluorenes	5	0.34
Phenanthrene	5	0.12
Anthracene	5	0.12
C1-Phenanthrenes/anthracenes	5	0.2
C2-Phenanthrenes/anthracenes	5	0.38
C3-Phenanthrenes/anthracenes	5	0.4
C4-Phenanthrenes/anthracenes	5	1.0
Fluoranthene	5	0.04
Pyrene	5	0.04
C1-Pyrene/fluoranthenes	5	0.078
Benz(a)anthracene	5	0.004
Chrysene	5	0.004
C1-Benzanthracenes/chrysenes	5	0.006
C2-Benzanthracenes/chrysenes	5	0.014
C3-Benzanthracenes/chrysenes	5	0.017
C4-Benzanthracenes/chrysenes ²	5	0.023
Benzo(b)fluoranthene	5	0.024
Benzo(k)fluoranthene	5	0.024
Benzo(e)pyrene	5	0.024
Benzo(a)pyrene	5	0.024
Perylene	5	0.024
Indeno(1,2,3-cd)pyrene	5	0.024
Dibenz(a,h)anthracene	5	0.024
Benzo(ghi)perylene	5	0.024

1. Compound may be reported as 2-, and 1-methylnaphthalene separately if requested.

2. Homologue range potentially not detectable in porewater by SPME methodology.

Sampling Guidelines: ASTM D-7673**Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Micro-extraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode**

This sheet provides procedures to be followed so that valid PAH concentrations in pore water samples are generated by this method. Field sampling methods are critically important to the laboratory test method. These procedures are recommendations by META Environmental, Inc. and are not part of ASTM D-7673 per se.

Sufficient wet sediment must be collected for each sample so that enough pore water can be extracted later for the test. Sediment samples should be WET. If the sediment samples are only moist, the laboratory may not be able to process the samples, or will need to perform an in-laboratory equilibration of the sediment with lab water to generate new “pore water”. There is an additional charge for this process.

Table 1 provides guidance on sample volumes, preservation, and handling.

The following sampling steps also should be conducted:

- Sample handling in the field should be described in the project-specific QAPP; however, the following guidelines can be applied:
 - Place the sediment from each location in a bucket or bowl.
 - Gently mix the sediment in the bucket or bowl first by hand using a chemically inert, stainless steel spoon or spatula.
 - Screen the sediment sample to remove oversized material. Materials such as shells, stones, pieces of wood, and vegetation are removed by hand and the sediment is press-sieved through a #5 mesh sieve (4 mm openings) with the spatula.
 - The sediment sample can then be homogenized using an electric drill-mounted mixing paddle or other suitable device.
- If the sieved slurry is to be stored or shipped before use, store in appropriate-sized, pre-cleaned amber glass jars with wide-mouthed PTFE-lined lids at 4°C, in the dark.
- Great care must be taken to clean the lid of the jar and the jar rim before capping with the lid to avoid leakage of the water during shipment. Wipe the jar rim and cap liner clean with a clean tissue prior to tightening the cap so that a good seal is formed. Pore water must not be lost and water from melted ice cannot be allowed to enter the sample jar during shipping and storage.
- For each sample, fill at least two 8-oz sample jars.
- Label each sample jar and complete the chain of custody.
- Ship in an ice chest with adequate ice to maintain 0 to 6°C. Store at the laboratory in the dark at 0 to 6°C.

Table 1. ASTM D-7673 Sample Volumes, Preservation, and Handling

Type of Sample	Matrix	Parameter	Minimum Quantity	Container Type(1)	Preservation (3)	Holding Time from Sample Date
Pore Water collected in the field	Water	Freely dissolved PAHs (parent compounds and estimate of alkylated PAHs)	2 x 40 mL VOA vials	glass	Cool to 4°C	24 hours
	Water	Dissolved Organic Carbon	1 liter	glass	Cool to 4°C	28 days
Pore Water collected from sediment in the laboratory	Sediment	Freely dissolved PAHs (parent compounds and estimate of alkylated PAHs)	2 x 8 oz wet sediment	glass	Cool to 4°C	28 days (4)
	Sediment	Dissolved Organic Carbon				
Bulk Sediment Analyses	Sediment	Total Organic Carbon	8 oz. (5)	glass	Cool to 4°C	28 days (2)
		Soot Carbon				28 days (2)
		Total PAHs parent & alkylated (34 compounds)				14 days (2)
		Percent Solids				28 days (2)
		Grain size	16 oz.	glass		NA
		pH	8 oz.	glass	Cool to 4°C	ASAP
		Ammonia			Cool to 4°C; adjust pH < 2	28 days (2)
Surface Water	Water	pH, Temperature, Conductivity, Salinity, DO, Turbidity	TBD	field	field	15 min.

(1) All glass jars must have Teflon-lined lids.

(2) Test to be initiated within 28 days of sample collection.

(3) Samples requiring thermal preservation must be maintained at 2° - 6° C.

(4) Pore water samples are generated in the laboratory from the sediment samples.

The holding time begins upon preparation of the pore water sample in the laboratory.

(5) Bulk sediment analyses subsamples can come from the same 8 oz jars as the pore water samples if sufficient mass is collected.

META Environmental, Inc.
Laboratory Standard Operating Procedure

SOP 7022 - 1

PARENT AND ALKYL POLYCYCLIC AROMATICS IN SEDIMENT PORE WATER
BY SOLID-PHASE MICROEXTRACTION AND GAS CHROMATOGRAPHY/MASS
SPECTROMETRY IN SELECTED ION MONITORING MODE

Summary of changes: All sections were either renamed or added as a new section, and renumbered to meet NELAP SOP requirements. References to external SOPs and the Laboratory Quality assurance Plan were inserted as appropriate.

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Author James A. Rouse

Quality Assurance David M. Manno

Laboratory Director David M. Manno

1.0 SCOPE AND APPLICATION

- 1.1 This method directly determines the concentrations of dissolved PAH concentrations in environmental sediment pore water, groundwater, and tap (drinking) water samples. This test entails the separation of pore water from PAH-impacted sediment samples, the removal of colloids, and the subsequent measurement of dissolved concentrations of 10 parent PAHs, two alkylated daughter PAHs, and 16 alkylated PAH homologues in the pore water samples. The following Polycyclic Aromatic Hydrocarbons (PAHs) can be determined by this method:

Analyte	CAS No ^a
Naphthalene	91-20-3
2-Methylnaphthalene	91-57-6
1-Methylnaphthalene	90-12-0
Acenaphthylene	208-96-8
Acenaphthene	83-32-9
Fluorene	86-73-7
Phenanthrene	85-01-8
Anthracene	120-12-7
Fluoranthene	206-44-0
Pyrene	129-00-0
Benz[a]anthracene	56-55-3
Chrysene	218-01-9
C1-Naphthalenes	NA
C2-Naphthalenes	NA
C3-Naphthalenes	NA
C4-Naphthalenes	NA
C1-Fluorenes	NA
C2-Fluorenes	NA
C3-Fluorenes	NA
C1-Phenanthrenes/Anthracenes	NA
C2-Phenanthrenes/Anthracenes	NA
C3-Phenanthrenes/Anthracenes	NA
C4-Phenanthrenes/Anthracenes	NA
C1-Fluoranthenes/Pyrenes	NA
C1-Benz(a)anthracenes/Chrysenes	NA
C2-Benz(a)anthracenes/Chrysenes	NA
C3-Benz(a)anthracenes/Chrysenes	NA
C4-Benz(a)anthracenes/Chrysenes	NA

^a: Chemical Abstract Registry Number

Regulatory methods using solvent extraction have not achieved the wide calibration ranges from nanograms to milligrams per liter and the required levels of detection in the nanogram per liter range. In addition, conventional solvent extraction methods require large aliquot volumes (liter or larger), use of large volumes of organic solvents, and filtration to generate the pore water. Solvent extraction entails the storage and processing of large volumes of sediment samples and loss of low molecular weight PAHs in the filtration and solvent evaporation steps.

This method can be used to determine nanogram to milligram per liter PAH concentrations in pore water. Small volumes of pore water are required for SPME

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extraction, only 1.5 mL per determination and virtually no solvent extraction waste is generated.

1.2 Lower molecular weight PAHs are more water soluble than higher molecular weight PAHs. Therefore, USEPA-regulated PAH concentrations in pore water samples vary widely due to differing saturation water solubilities that range from 0.2 µg/L for indeno[1,2,3-cd]pyrene to 31,000 µg/L for naphthalene. This method can accommodate the measurement of milligram per liter concentrations for low molecular weight PAHs and nanogram per liter concentrations for high molecular weight PAHs.

1.3 This method can achieve the required detection limits, which range from approximately 0.01 µg/L, for high molecular weight PAHs, to approximately 3 µg/L for low molecular weight PAHs.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Pore water is separated from wet sediment samples by centrifugation and supernatant collection. The groundwater and tap water samples begin preparation with the colloid removal step. Colloids are removed from the separated pore water, groundwater, and tap water samples by flocculation with aluminum potassium sulfate (alum) and sodium hydroxide. A second flocculation and centrifugation step, followed by supernatant collection completes the colloid removal.

2.2 The PAHs are determined using solid-phase microextraction (SPME) followed by Gas Chromatography/Mass Spectrometry analysis in selected ion monitoring (SIM) mode. Either the use of an autosampler, or a manual approach can be used to perform the SPME extraction and the subsequent injection of collected analytes into the GC/MS. Isotopically labeled analogs of the target compounds are introduced prior to the extraction, and are used as quantification references.

2.3 The mass spectrometer is operated in the SIM mode for the molecular ions of the target PAHs and d-PAHs to achieve low limits of detection. Analyte concentrations are quantified by two methods: 1) Parent PAHs (i.e. unsubstituted PAHs) for which an exact deuterated analog is not included in the internal standard mix are quantified by reference to a deuterated analog of a PAH with the same number of rings as the analyte, or 2) PAHs for which an exact deuterated analog is included in the internal standard mix are quantified by isotope dilution.

2.4 Test Method Options - Either the use of an autosampler, or a manual approach can be used to perform the SPME extraction and the subsequent injection of collected analytes into the GC/MS. An autosampler (Leap Technologies Compi-Pal or equivalent) is much preferred over the manual method because: (1) the autosampler yields lower and more reproducible blanks, (2) the manual method requires the use of a stir bar that can cause sample cross-contamination, and, (3) the manual method is highly labor-intensive and requires multiple timed manipulations per analysis leading to operator fatigue and resultant errors, and (4) the autosampler reduces the technician time required to prepare samples for a 24-hour run sequence to approximately 3 hours, while the manual method requires 24 hour operator attendance. Therefore, the method procedures are written

assuming the use of an autosampler, with modifications to the autosampler procedures listed for the manual method.

2.4.1 Autosampler Method

2.4.1.1 Pore Water Separation and Preparation: Pore water is separated from wet sediment samples by centrifugation and supernatant collection. The groundwater and tap water samples begin preparation with the colloid removal step. Colloids are removed from the separated pore water, groundwater, and tap water samples by flocculation with aluminum potassium sulfate (alum) and sodium hydroxide. A second flocculation and centrifugation, followed by supernatant collection completes the colloid removal. The prepared water samples are then split into the required number of replicate aliquots (1.5 mL each) and placed into silanized glass autosampler vials. The 8 perdeuterated PAH internal standards (d-PAHs) are then added immediately. All of the water preparation steps beginning with the centrifugation and ending with the addition of d-PAH internal standards should be conducted continuously and in the minimum amount of time possible.

The SPME fiber should be cleaned at the beginning of each sampling set (and after very contaminated samples) for one hour by placing in the cleaning chamber under helium flow at 320° C. This can conveniently be performed while the water samples are being prepared.

2.4.1.2 Solid-Phase Microextraction: The SPME extraction of the water samples is performed using a commercially available (available from Sigma-Aldrich, formerly Supleco, or equivalent) 7 µm film thickness polydimethylsiloxane (PDMS)-coated fused silica fiber for 30 min while the water sample is mixed by the precession of the autosampler mixing chamber at a rate of 250 revolutions per minute. The target PAHs and d-PAH internal standards adsorb to the nonpolar PDMS phase at equivalent rates. The use of the d-PAHs (i.e., isotopic dilution) to quantitate the target PAHs compensates for variations in equilibrium partitioning and kinetics.

2.4.1.3 GC/MS SIM Analysis: Following the sorption period, the SPME fiber is immediately desorbed to a GC/MS injection port in the splitless mode at 320° C for 5 min. The GC/MS system specified uses a 60 meter narrow-bore (250 µm i.d.) HP5-MS or equivalent capillary column to achieve high resolution for PAHs. Following the 5 minute desorption period, the SPME fiber is inserted into the cleaning port and additionally cleaned for 15 minutes under helium flow at 320° C. At the end of the cleaning period, sorption of the next water sample is begun.

2.4.2 Manual Method

2.4.2.1 Alternate Procedures for Manual Method: Samples are prepared as for the autosampler method, except that a small Teflon-coated stir bar is placed in the silanized autosampler vial prior to adding the water and d-PAH internal standard solution. A new stir bar should be used for each sample, calibration standard, and blank to avoid cross-contamination caused by carryover on the stir bar. To perform the SPME step, the vial is set on a stir plate and the stirring rate adjusted so that no large vortex is formed. The SPME fiber should be inserted into the water so that the entire 1-cm active length is exposed to the water sample, but not so low

that the fiber comes into contact with the stir bar or that the metal needle sheath contacts the water. All time sequences should be the same as specified for the autosampler method. A spare GC split/splitless injection port at 320° C and under helium flow can be used for the 15 minute cleaning step between samples as well as for the initial 1-hour cleaning step at the beginning of each experimental day.

2.5 This method includes specific calibration, sample analysis, and quality control steps that supersede the general requirements provided in Method 8000.

3.0 DEFINITIONS

3.1 Calibration Standard – A solution prepared from a secondary standard and/or stock solution and used to calibrate the response of the instrument with respect to analyte concentration.

3.2 Calibration Verification Standard (VER) – The mid-point calibration standard (CS3) that is analyzed daily to verify the initial calibration.

3.3 CS1, CS2, CS3, CS4 – Shorthand notation for calibration standards.

3.4 Data Acquisition Parameters – Parameters affecting the scanning operation and conversion of the analytical signal to digitized data files. These include the configuration of the ADC circuitry, the ion dwell time, the MID cycle time, and acquisition modes set up for the method. Examples of acquisition modes for the HP5973 include SIM mode, and Low Mass Resolution Mode.

3.5 Performance Limit – The Performance Limit for each individual PAH is defined as the concentration of an individual PAH that would yield 1/34 of a toxic unit. For the performance limits of each individual PAH refer to Table 4.

3.6 Deuterated PAH (d-PAH) – Polycyclic aromatic hydrocarbons in which deuterium atoms are substituted for all hydrogens (i.e., perdeuterated). In this method, d-PAHs are used as internal standards.

3.7 GC – Gas chromatograph or gas chromatography

3.8 HRGC – High resolution GC

3.9 LRMS – Low resolution MS

3.10 Internal Standards – Isotopically labeled analogs (d-PAHs) of the target analytes that are added to every sample, blank, quality control spike sample, and calibration solution. They are added to the water samples immediately after completing the flocculation step and transferring the water aliquot to the autosampler vial, and immediately after adding the calibration PAH solution to water calibration standards, but before SPME extraction. The internal standards are used to calculate the concentration of the target analytes or estimated detection limits.

3.11 Laboratory Blank – See Method Blank.

3.12 Method Blank – An aliquot of reagent water that is extracted and analyzed along with the samples to monitor for laboratory contamination. Blanks should consistently meet concentrations at or less than one-third of the Performance Limits for individual PAHs

stated in Table 4. Alternatively, if the PAH concentrations calculated from the water blank immediately preceding the test samples are <20% of the test sample concentrations, the blank is acceptable.

3.13 Low Calibration Level (LCL) – The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

3.14 High or Upper Calibration Level (UCL) – The concentration or mass of analyte in the sample that corresponds to the highest calibration level in the initial calibration. It is equivalent to the concentration of the highest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

3.15 MS – Mass spectrometer or mass spectrometry.

3.16 PAH – Polycyclic aromatic hydrocarbon, or alternately, polynuclear aromatic hydrocarbon.

3.17 Percent Difference (%D) – The difference between the analyzed concentration and expected concentration, expressed as a percentage of the expected concentration.

3.18 Relative Response Factor (RRF) – The empirically determined ratio between the area ratio (analyte to internal standard) and the unit mass of analyte in the calibration standard (area ratio/ng) for the two alkyl PAHs and the parent PAHs.

3.19 Selected Ion Monitoring (SIM) – A mode of operation for the mass spectrometer in which specific ions are monitored. This mode of operation differs from the full scan mode, in which the MS acquires all ions within a range. Because the spectrometer is monitoring fewer ions in the SIM mode, more acquisition (dwell) time is possible for each ion. This results in greater instrument sensitivity for the selected ions. Spectral scanning and library searching, used for tentatively identified compounds, are not supported in this mode.

3.20 Signal-to-Noise Ratio – The ratio of the mass spectrometer response of a GC peak to the background noise signal.

4.0 INTERFERENCES

4.1 Non-target hydrocarbons can cause peaks on selected ion current profiles (SICPs) intended for other PAHs. Analysts should be familiar with both parent and alkyl PAH analyses in complex environmental samples.

4.2 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using PVC gloves, powdered gloves, or gloves with measurable levels of phthalates. The use of high purity reagents and solvents helps minimize interference problems.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the mass spectrometer must be vented to the laboratory hood exhaust system or must pass through an activated charcoal filter.

5.3 Primary Materials Used: Table 2 contains a summary of the primary hazards listed in the MSDS. A complete list of materials used in the method can be found in the reagents and standards section. Practitioners must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 EQUIPMENT AND SUPPLIES

This section does not list common laboratory glassware (e.g., beakers and flasks).

The mention of trade names or commercial products in this method is for illustrative purposes only, and does not constitute a USEPA endorsement or exclusive recommendation for use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance for the intended application has been demonstrated and documented.

- 6.1** Centrifuge capable of sustaining 1000 g with cups for securing 40 mL and 20 mL vials.
- 6.2** SPME fiber holder compatible with 7- μ m SPME fiber and compatible with either the autosampler or the manual method.
- 6.3** SPME fused silica fibers coated with 7 μ m film thickness polydimethylsiloxane (PDMS) from Sigma-Aldrich (formerly Supelco®) or equivalent.
- 6.4** PTFE coated stir bars (stir fleas) of a size effective for stirring 1.5 mL water without vortexing (for manual method only).
- 6.5** Magnetic stir plate (for manual method only).
- 6.6** SPME holder stand (for manual method only) or GC/MS autosampler capable of SPME extraction and injection (LEAP Technologies Combi-Pal or equivalent).
- 6.7** Cleaning port, capable of purging SPME fibers in a helium-swept atmosphere at 320° C.
- 6.8** 40 mL vials with Teflon-lined caps.
- 6.9** 20 mL vials with Teflon-lined caps.

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- 6.10** Silanized 2.0 mL autosampler vials.
- 6.11** GC/MS Analysis
 - 6.11.1 Gas Chromatograph – Shall have split/splitless injection port for capillary column, temperature program with isothermal hold.
 - 6.11.2 GC column – 60m x 0.25 mm ID x 25 μ m film thickness HP5-MS or equivalent.
 - 6.11.3 Inlet liner 2 mm i.d. silanized glass.
 - 6.11.4 GC inlet 320° C, splitless mode.
 - 6.11.5 Oven program: Isothermal 5 minute hold at 40° C. Ramp at 50° C/minute to 110° C, followed by a temperature ramp of 12° C/minute to 320° C (Hold for 10 min).
 - 6.11.6 Mass Spectrometer – Electron impact ionization with the ionization energy optimized for best instrument sensitivity (typically 70 eV), stability and signal to noise ratio. Shall be capable of repetitively selectively monitoring at least 12 m/z's during a period of approximately 1 second and shall meet all manufacturers' specifications.
 - 6.11.7 GC/MS Interface – The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within 1 cm of the ion source but does not intercept the electron or ion beam.
 - 6.11.8 Data System – Capable of collecting, recording, and storing MS data.

7.0 REAGENTS AND STANDARDS

- 7.1** Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
- 7.2** Reagent water: Water that meets the purity specifications of Type I or Type II water presented in ASTM D1193, HPLC-grade water, deionized water, free of the analytes of interest.
- 7.3** Internal standard stock solution. A dichloromethane solution of d-PAH internal standards used for preparing spiking solutions by dilution into acetone. (See Section 10.3.)
- 7.4** Internal standard spiking solution. A dilution of the internal standard stock solution in acetone used to spike d-PAH internal standards into all sample, calibration, and blank water vials. (See Section 10.3.)
- 7.5** Calibration stock solution. A dichloromethane solution of PAHs used for preparing calibration standards. (See Section 10.3.)
- 7.6** Calibration Spiking Solutions. A series of solutions prepared by diluting the calibration stock solution with acetone. (See Section 10.3.)

- 7.7** Calibration Standards. Prepared by adding internal standard and calibration spiking solutions in reagent water. (See Section 10.3.)
- 7.8** Acetone
- 7.9** Dichloromethane (DCM)
- 7.10** Sodium Hydroxide (NaOH)
- 7.11** Aluminum Potassium Sulfate Dodecahydrate ($\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$).
- 7.12** Alum Solution: Add 20g ($\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) to 80 mL reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 8.1** Collect the sediment, groundwater, and tap water samples in accordance with the practices outlined in ASTM D 3370 and Specification D 1192, as applicable.
- 8.2** Prior to shipment, the sediment samples should be mixed well. Sieve the slurry of sediment and site water through a 2-mm screen to remove debris. If the sieved slurry is to be stored or shipped before use, store in 250 mL to 1L jars with PTFE-lined lids. Great care must be taken to clean the lid of the jar before capping with the lid to avoid leakage of the water during shipment. Groundwater and tap water samples should be stored in 250 mL to 1L glass bottles with PTFE-lined caps.
- 8.3** Ship samples in an ice chest with adequate ice to maintain 0-6° C. Store at the laboratory in the dark at 0-6° C.

9.0 QUALITY CONTROL

- 9.1** Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and directions for those that will implement the project and assess the results. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference and inspection.
- 9.2** The laboratory using this test must perform an initial demonstration of laboratory capability. Analyze seven replicates on an initial demonstration of performance (IDP) solution. The IDP solution is a reagent water or field sample matrix solution fortified with the method analytes and internal standards at known concentrations, and prepared from a different source than that used to prepare the calibration standards. Ideally, the IDP solution should be prepared by an independent source. The mean and standard deviation of the seven values should then be calculated and compared to the test method single operator precision.

- 9.3 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are free from contaminants and interferences. This is accomplished through the analysis of extraction and analytical blanks. The following acceptance criterion will be used for extraction and analytical blanks: Analyzed between every sample to monitor the baseline. Target analytes must not be detected above 1/3 of the Performance Limits or > 20% of the associated sample result(s).

The following corrective action will be adopted for extraction and analytical blanks: Locate the source of the contamination; correct the problem. Re-extract and reanalyze the associated samples that are less than ten times the level of the contaminants present in the method blank.

- 9.4 The following acceptance criteria will be used for initial calibration: (i) The signal to noise (S/N) ratio for the GC signals present in every selected ion current profile (SICP) must be $\geq 10:1$ for the labeled internal standards and calibration compounds; (ii) The percent relative standard deviation (RSD) for the mean area ratio/ng for labeled internal standards and the calibration compounds must be less than 30% for high molecular weight PAHs and less than 25% for low molecular weight PAHs, and the $r^2 > 0.99$. The calibration curve must not be forced through the origin; (iii) The number of calibration standards may be reduced from four to three based on the criteria in Section 10.4.1 of this procedure.

The following corrective action will be adopted for a nonconforming initial calibration: (i) Initial calibration must be re-established if the RSD(s) exceed the limit(s); (ii) The calibration will not be re-established in response to a nonconforming RSD if the sample results are less than the PQL.

- 9.5 The following acceptance criteria will be used for daily duplicate calibration verifications: (i) The S/N ratio for the GC signals present in every SICP must be $\geq 10:1$ for the labeled internal standards and the calibration compounds; (ii) The percent differences for the measured area ratio/ng of all analytes must be within $\pm 25\%$ for high molecular weight PAHs and less than $\pm 20\%$ for low molecular weight PAHs of the mean values established during the initial calibration.

The following corrective action will be adopted for nonconforming daily duplicate calibration verifications if the first acceptance criterion is not satisfied: a new initial calibration curve must be established before sample extracts can be analyzed.

- 9.6 The signal to noise (S/N) ratio for the GC signals present in every selected ion current profile (SICP) must be $\geq 3:1$ for target compounds in environmental samples and $\geq 10:1$ for the labeled internal standards.

The following corrective action will be adopted for signal to noise ratio: Reanalyze the sample unless obvious matrix interference is present.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 To prepare the apparatus, set up the GC system using the following parameters.

- 10.1.1 GC Column Agilent HP-5MS column (0.25 µm film thickness, 0.25 mm ID) or equivalent.
- 10.1.2 Inlet liner 2-mm i.d. silanized glass.
- 10.1.3 GC Inlet 320° C, splitless mode.
- 10.1.4 Oven program: Isothermal 5 minute hold at 40° C. Ramp at 50° C/minute to 110° C, followed by a temperature ramp of 12° C/minute to 320°C. (Hold for 10 min.)
 MS Quad Temperature: 150° C, maximum 200° C
 MS Source Temperature: 230° C, maximum 250° C
- 10.2 Set up SIM Groups**
 - 10.2.1 Set up a SIM program with the necessary ions to acquire all the PAHs using the ion groups shown in Table 4 and 25 msec dwell time per ion.
 - 10.2.2 Update the expected retention times in the method section of the quantitation software using the d-PAH internal standards of previous runs as relative retention time markers..
- 10.3 Analyze Initial Calibration:**
 - 10.3.1 Prepare stock solutions of PAHs and internal standard stock solutions of d-PAHs at approximately the concentrations shown in Table 5. These concentrations were based on the PAH distributions previously determined in 120 sediment pore water samples. Stocks are prepared in DCM. Spiking solutions are prepared by dilution of intermediate stocks in acetone. For calibration solutions, spiking solutions are added to reagent water.
 - 10.3.1.1 Prepare calibration standard spiking solutions. These are prepared by adding acetone to the stock to give the calibration solution concentrations (CS1-CS4), as described below.
 - 10.3.1.1.1 For CS1, take 5 µL stock to 100 mL in acetone.
 - 10.3.1.1.2 For CS2 take 50 µL to 100 mL in acetone.
 - 10.3.1.1.3 For CS3, take 25 µL to 10 mL in acetone.
 - 10.3.1.1.4 For CS4, take 100 µL to 10 mL in acetone.
 - 10.3.1.2 Spike 4 µL of each calibration solution into 1.5 mL of reagent water to give a calibration series with the low calibration limits (LCLs) and upper calibration limits (UCLs) shown in Table 3. Spike 10 µL of internal standard spiking solution at the concentrations shown in Table 5 into each vial.
 - 10.3.1.3 Extract and analyze the calibration series.

10.3.1.3.1 Extract and analyze two water blank solutions.

10.3.1.3.2 Extract and analyze the water calibration solutions, as described in Sections 11.4 and 11.5. Begin with the CS1-spiked sample, followed by sequentially more concentrated calibration standards. Follow by two water blanks.

10.3.1.4 Calculate the performance parameters for the calibration.

10.3.1.4.1 Generate ion chromatograms for the masses listed in Table 4 that encompass the expected retention windows of the target analytes. Integrate the selected ion current profiles of the quantitation ions shown in the table.

10.3.1.4.2 Calculate the area ratio (analyte peak area divided by internal standard peak area) per unit mass of analyte, using the area of the appropriate internal standard listed in Table 4.

Quantitative calculations are based on a comparison of the area ratio per ng from the calibration and sample waters. The area ratio per ng is calculated for calibration runs by dividing the calibration peak area by the peak area of its most closely associate d-PAH internal standard (the deuterated parent PAH, in most cases), and dividing this result by the ng of the calibration PAH present in the vial (i.e., its mass in the vial, not its concentration). Calibration standards are given in Table 5.

(area ratio/ng) = [(peak area cal. std)/(peak area d-PAH)]/(mass of std in cal vial)

10.3.1.4.3 Calculate the mean area ratio/ng. The mean relative response factor for these duplicate daily calibration standards should agree with those from the 4-point (or 3-point) standard curve within 20% for the two- and three-ring PAHs, and within 25% for the four-ring PAHs. No sample data will be reported if these calibration criteria are not met. Calculate the mean area ratio/ng and the standard deviation of the relative response factors for each calibration standard solution using the following equations:

$$\overline{\text{area ratio/ng}} = \frac{1}{n} \sum_{i=1}^n (\text{area ratio/ng})_i$$

Where:

(area ratio/ng)_i = area ratio/ng calculated for calibration solution “i” using the equation in Section 10.3.1.4.2.

n = The number of calibration points in the curve.

10.3.1.4.3.1 Calculate the percent relative standard deviation.

$$\% \text{RSD} = \frac{\text{SD}}{\text{area ratio/ng}} \times 100$$

Where:

$\overline{\text{area ratio/ng}}$ = Mean area ratio/ng calculated above.

SD = The sample standard deviation of the replicate area ratio/ng values used to calculate the mean area ratio/ng.

10.4 Criteria for acceptable initial calibration. Prior to analyzing any samples, the standard curves are prepared using the identical analysis procedures as used for sample waters. To be acceptable, the linearity of each PAH standard curve should be $r^2 > 0.99$, and the relative response factor per ng for each concentration should show a relative standard deviation of <25% for two- to three-ring PAHs, and <30% for four-ring PAHs. If acceptable initial calibration is not achieved, identify the root cause, perform corrective action, and repeat the initial calibration. If the root cause can be traced to an abnormal disruption of an individual acquisition (e.g. injector malfunction) repeat the individual analysis and recalculate the percent relative standard deviation. If the calibration is acceptable, document the problem and proceed; otherwise repeat the initial calibration.

10.4.1 Because of the large range of calibration concentrations required, the wide range of water solubilities of the individual PAHs, and the desire to require only one stock calibration solution, some PAHs may only have a three point linear calibration curve that meets the above criteria. This is most likely to occur for the higher molecular weight PAHs, because the dilution of lowest calibration standard is likely to be below detection limits for many labs (and is also below the required detection limits needed for the method, so it does not negatively impact the analyses). In such cases, the lowest calibration standard is ignored, and the "J" level adjusted appropriately. Less frequently, the highest concentrations of the lowest molecular weight PAHs may exceed the linear dynamic range of the GC/MS response. In such cases the laboratory should investigate lowering the MS multiplier voltage to autotune voltage or slightly below and rerun the calibration curve. If the highest calibration standard still exceeds the detector linearity, it is acceptable to reject the highest concentration for those specific PAHs (and adjust the "E" value accordingly), as long as a minimum of a three-point standard curve is generated for each PAH.

It is recommended that a 4- (or 3-) point initial calibration be established every two weeks, when continuing calibration criteria are not met, or when service is performed on the GC/MS instrument system.

10.4.2 The signal to noise ratio (S/N) for the GC signals present in every selected ion current profile (SICP) must be $\geq 10:1$ for the labeled internal standards and unlabeled calibration compounds.

10.5 Continuing calibration is performed daily at the beginning of a 24-hour period. The injection of the first continuing calibration begins the 24-hour window, within which all pore water samples must be injected. Duplicate daily standards are analyzed.

10.5.1 Into 1.5 mL of reagent water, add 4 µL of the CS3 spiking solution and 10 µL of the d-PAH internal standards.

10.5.2 Analyze duplicate vials of the Calibration Standard Solution CS3. Use the same data acquisition parameters as those used during the initial calibration. Check for GC resolution and peak shape. If peak shape or retention times are unacceptable, perform column and injector maintenance. If this fails to correct the problem, the column must be replaced and the calibration repeated.

10.5.3 Criteria for Acceptable Daily Calibration Check. The criteria listed below for acceptable calibration must be met at the beginning of each 24-hour period that samples are analyzed. The mean relative response factor for the duplicate daily calibration standards should agree with those from the 4-point (or 3-point) standard curve within 20% for the two- and three-ring PAHs, and within 25% for the four-ring PAHs. No sample data will be reported if these calibration criteria are not met. If the continuing calibration criteria are not met, identify the root cause, perform corrective action and repeat the continuing calibration. If the second consecutive continuing calibration does not meet acceptance criteria, additional corrective action must be performed.

10.5.4 The signal to noise ratio (S/N) for the GC signals present in every selected ion current profile (SICP) must be $\geq 10:1$ for the labeled internal standards and unlabeled calibration compounds.

10.6 Method blanks are prepared and analyzed daily in duplicate following the continuing calibration and between analysis of replicate sets of the same pore water sample. See Section 10.6.2.2.

10.6.1 For each method blank, add 10 µL of the d-PAH internal standards solution into 1.5 mL of reagent water.

10.6.2 Two types of sources of background PAHs must be considered. For the higher molecular weight PAHs, typical GC/MS criteria for signal to noise are appropriate, since their detection limits are normally controlled by GC/MS sensitivity. However, for lower molecular weight PAHs, atmospheric contaminants can cause significant background peaks. This problem is most likely to be significant in urban areas impacted by atmospheric PAHs (e.g, from diesel exhaust), and with laboratories using manual techniques, rather than the SPME autosampler.

10.6.2.1 Background PAHs from Ambient Air – Concentrations of each PAH in the water blanks should be calculated in the same manner as a sample. Should the blank prior to the subsequent water sample have any detectable background concentration greater than 1/3 of the Performance Limits given in Table 4, the analyses should not continue until the fiber is sufficiently cleaned as demonstrated by a clean water blank. The mean of the calculated concentrations of the PAHs in the blanks analyzed immediately before and immediately after sample waters should be subtracted from the sample water concentrations.

10.6.2.2 Carryover from Highly Contaminated Samples – Carryover blanks are analyzed between each new pore water sample (not including replicates). Significant carryover can occur if the previous sample was highly contaminated. Should the blank prior to the subsequent water sample have any detectable background concentrations more than 1/3 of the Performance Limits given in Table 4, the analyses should not continue

until the fiber is sufficiently cleaned as demonstrated by a clean water blank. Alternatively, if the concentrations determined in the blanks are less than 20% of those found in the related sample, the data can be accepted.

11.0 PROCEDURE

11.1 At the laboratory, store samples and extracts in the dark at 0 to 6°C.

11.2 Holding times

11.2.1 Pore waters must be generated within 28 days of sediment sample collection.

11.2.2 Pore waters must be generated and flocculated as quickly as possible. Pore water, groundwater, and tap water samples must be immediately spiked with 10 µL of d-PAH solution following flocculation.

11.2.3 Solid phase micro-extraction must be completed within 24 hours of flocculation for pore water, groundwater, and tap water samples.

11.3 Generation of pore water from sediment samples.

Stir the slurry and transfer approximately 40 mL (containing a solids and liquids in proportion to the slurry provided) to a clean 40 mL vial. Cap the vial with a PTFE-lined cap. Place the vials in a centrifuge. Spin for 30 minutes at 1000 g. Using a new, graduated serological pipette, transfer 10 mL of the supernatant to a new 20 mL vial.

11.4 Flocculation of pore water, groundwater, and tap water samples.

11.4.1 Add the working alum solution (see Section 7) to each vial of water (and QC samples). The volume of the alum solution should be 1/40th of the sample volume. After the addition, swirl the vial for several rotations to incorporate the solution.

11.4.2 Add 3-5 drops of NaOH working solution (see Section 7) to each vial. Swirl to incorporate the NaOH.

11.4.3 Shake the vial for 15 seconds.

11.4.4 Centrifuge for 30 minutes at 1000 g.

11.4.5 Collect the supernatant into a clean 20 mL vial.

11.4.6 Repeat Sections 11.4.1 through 11.4.5 once.

11.4.7 Immediately transfer 1.5 mL aliquots to new silanized autosampler vials and immediately add the internal standard solution as described below. Vials are weighed before and after adding the water sample to determine the exact sample water mass.

11.5 Extraction and analysis of flocculated pore water, groundwater, and tap water samples.

11.5.1 Split the prepared water samples into the required number of replicate samples, placing 1.5 mL aliquots of each into a new silanized glass autosampler vial. For QC samples, add 1.5 mL of reagent water.

Note: The SPME fiber should be cleaned at the beginning of each sampling set (and after very contaminated samples) for one hour by placing in the cleaning chamber under helium flow at 320° C. This can conveniently be performed while the pore waters are being prepared.

11.5.2 Immediately add 10 µL of the d-PAH solution to each sample and QC sample.

Note: All of the water preparation steps beginning with the centrifugation and ending with the addition of d-PAH internal standards should be conducted continuously and in the minimum amount of time possible.

11.5.3 Load the autosampler following the recommended analytical sequence in Table 5. Verify the sequence against documented sequence following the loading process.

11.6 The recommended analytical sequence described in Table 5 is based on a 24-hour "clock."

11.6.1 Two calibration verification standards are analyzed (100 min.). The sequence begins with analysis of the first continuing calibration standard.

11.6.2 Analyze two method blanks (50 min. each).

11.6.3 Analyze pore water samples (in duplicate at a minimum) (50 min. each).

12.0 Data Analysis and Calculations

12.1 Generate ion chromatograms for the masses listed in Table 1 that encompass the expected retention windows of the target analytes. Integrate the selected ion current profiles of the quantitation ions shown in the table.

Qualitative identification criteria for individual analytes: For a gas chromatographic peak to be identified as a target analyte, it must meet all of the following criteria.

12.1.1 The quantitation ion must be present, with a signal-to-noise ratio of at least 3:1 for environmental samples.

12.1.2 The relative retention time (RRT) of the parent PAHs (and the 2- and 1-methylnaphthalene compounds) compared to the RRT for the labeled-standards must be within ± 3 seconds of the relative retention times obtained from the continuing calibration (or initial calibration if this applies). The retention time (RT) of the analyte must be no more than 5 seconds before the expected RT of the first isomer in the homologue, based on the continuing windowing solution analysis.

12.2 Quantitation for Target Analytes

Sample water concentrations are calculated by dividing the peak area of the sample peak by the peak area of its d-PAH internal standard, and then dividing the result by the calibration area ratio per ng, and dividing that result by the sample water weight.

$$\text{Concentration (ng/mL)} = \frac{(\text{area sample peak}) / (\text{area d - PAH peak})}{(\text{area ratio per ng cal. std}) / (\text{sample weight})}$$

The mean calibration area ratio per ng values from the daily calibration runs is used for sample concentration calculations (assuming QA/QC checks with the full calibration curve meet criteria).

Note: The two methylnaphthalene isomers are individual alkyl peaks and are treated as parent PAHs in the calculations.

12.2.1 If no peaks are present at a signal to noise value ≥ 3 to 1 in the region of the ion chromatogram where the compounds of interest are expected to elute, report the result as "Not Detected" (i.e., ND) at the reporting limit.

12.2.2 Depending on project objectives, the results may be reported to Performance Limits or estimated detection limits (EDLs).

If project-specific guidance requires analysis-specific EDLs, calculate the detection limit for that compound according to the following equation:

$$\text{Estimated Detection Limit} = \frac{N \times 2.5}{H_{is} \times (\text{area ratio} / \text{ng})}$$

Where:

N = height of peak to peak noise of quantitation ion signal in the region of the ion chromatogram where the compound of interest is expected to elute
H_{is} = peak height of quantitation ion for appropriate internal standard
area ratio/ng = mean area ratio/ng of compound obtained during daily calibration

If project-specific guidance requires total toxic units (TTU) to be reported, calculate the detection limit for that compound according to the following equations:

$$TU_c = C_{tu} \times \text{result} (\text{ng} / \text{mL})^{-1}$$

$$\text{Total Toxic Units (TTU)} = \sum_1^n TU_c$$

Where:

TU_c = Toxic Unit concentration for each individual compound or homolog (ng/mL).
C_{tu} = concentration for one Toxic Unit (ng/mL), see Table 4.
result = individual pore water result for a compound (ng/mL).
TTU = total toxic units for all compounds.

12.2.3 Flag all compound results in the sample which were estimated below the lowest calibration level with a "J" qualifier

12.2.4 Flag all compound results in the sample which were estimated above the upper calibration level with an "E" qualifier.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance goals for users of the methods. Instead, performance goals should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method.

13.2 Tables 6 through 9 present precision and bias data from multiple laboratories for a spiked groundwater, tap water, and two sediment pore water matrices. The PAH-impacted sediment samples were collected from a manufactured gas plant (MGP) site and a smelter site. These data are provided for guidance purposes only.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in the laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington D.C. 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

15.1 The USEPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Section 14.2.

15.2 Some of the reagents and solutions used in this method as well as the effluent from the chromatograph contain PAHs and should be handled and disposed of in an approved manner.

16.0 REFERENCES

1. American Society for Testing Methods (ASTM), D2777-06, Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water.
2. American Society for Testing Methods (ASTM), D5847-02, Standard Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis.
3. Hawthorne, S.A., Grabanski, C.B., Miller, D.J., Kreitinger, J.P., Environ. Sci. Technol. 2005, 39, 2795-2803, Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values.
4. Thal, D.I., Standard Operating Procedure KNOX-ID-0019. Preparation of Flocculated Pore Waters and Analysis of Parent and Alkyl Polycyclic Aromatic Hydrocarbons by Isotope Dilution Solid Phase Microextraction/Gas Chromatography/Mass Spectrometry. (SPME/GC/MS) (Hawthorne Method). ©Severn Trent Laboratories. 2006.
5. Hawthorne, Steven B., Grabanski, Carol B., Miller, David J., Environmental Toxicology and Chemistry 2006, 25, 2901-2911, Measured Partitioning Coefficients for Parent and Alkyl Polycyclic Aromatic Hydrocarbons in 114 Historically contaminated Sediments: Part I, K_{OC} Values."
6. American Society for Testing Methods (ASTM), D7673-07, Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode1,

17.0 TABLES AND FIGURES

The pages to follow contain the tables and figures referenced by this method.

TABLE 1 Relative Response Factors^A

Analyte	SPME-GC/MS RRF ^B versus Parent	Basis for Performance Limit ^C
Naphthalene	1.00	B
2-Methylnaphthalene ^D	1.00	B
1-Methylnaphthalene	1.00	B
C2-Naphthalenes	1.44	B
C3-Naphthalenes	0.88	B
C4-Naphthalenes	0.71	C
Acenaphthylene	1.00	B
Acenaphthene	1.00	B
Fluorene	1.00	B
C1-Fluorenes	0.73	B
C2-Fluorenes	0.59	B
C3-Fluorenes	0.35	S
Phenanthrene	1.00	B
Anthracene	1.00	B
C1-Phenanthrenes/Anthracenes	0.57	B
C2-Phenanthrenes/Anthracenes	0.32	B
C3-Phenanthrenes/Anthracenes	0.29	B
C4-Phenanthrenes/Anthracenes	0.12	S
Fluoranthene	1.00	B
Pyrene	1.00	B
C1-Fluoranthenes/Pyrenes	0.51	C
Benz[a]anthracene	1.00	B
Chrysene	1.00	B
C1-Chrysenes/Benz[a]anthracenes	0.62	C

^A From Hawthorne, S. B., Grabanski, C.B., Miller, D .J., and Kreitinger, J. P., "Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," *Environmental Science Technology*, 39, 2005, pp. 2795-2803.

^B All relative response factors are based on the SPME-GC/MS peak area per ng of the alkyl PAH in a water standard compared to that of its parent PAH as determined by SPME followed by GC/MS. When several isomers were available, (for example, C2-naphthalenes), the mean relative response factor is reported. The relative response factors of alkyl PAHs for which no standards were available were estimated based on the closest analogous alkyl PAH as described in reference 2.1.

^C Performance limits were determined as 3 times the background concentrations from the SPME fiber based on the analysis of water blanks ("B"), the lowest calibration standard which consistently yielded a signal to noise ratio of at least 3:1 ("C"), or (for when no calibration standard was available) for the lowest concentrations consistently found in pore water samples with a signal to noise ratio of at least 3:1 ("S"). Detection limits for alkyl PAHs are based on a single isomer.

^D Alkyl PAHs used to determine the SPME-GC/MS relative response factors including alkyl naphthalenes (1-methyl-, 2-methyl-, 1,2-dimethyl-, 1,3-dimethyl-, 1,8-dimethyl-, 2,7-dimethyl-, 1-ethyl-, 2-ethyl-, 1,4,5-trimethyl-, 2,3,5-trimethyl-, and 2-isopropyl-), 1-methylfluorene, 2-methyl- and 9-methylanthracene, 1-methyl-, 2-methyl-, and 3-methylphenanthrene, 9,10-dimethylanthracene, 2-ethylanthracene, 2-tertbutylanthracene, 1-methyl-7-isopropylphenanthrene, 1-methylpyrene, 7-methylbenz[a]anthracene, and 7,12-dimethylbenz[a]anthracene.

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TABLE 2 Toxic Unit Factors and Performance Limits^A

Analyte	Added d-PAH Internal Standard	d-PAH Internal Std. for Calculation	SPME-GC/MS RRF versus Parent	Conc. for One Toxic Unit, C _{TU} (ng/mL)	Performance Limit (ng/mL)
Naphthalene	A	A	1.00	193.47	5.69
2-Methylnaphthalene		B	1.00	81.69	2.40
1-Methylnaphthalene	B	B	1.00	81.69	2.40
C2-Naphthalenes		A	1.44	30.24	0.89
C3-Naphthalenes		A	0.88	11.10	0.33
C4-Naphthalenes		A	0.71	4.05	0.12
Acenaphthylene		C	1.00	306.85	9.03
Acenaphthene	C	C	1.00	55.85	1.64
Fluorene	D	D	1.00	39.30	1.16
C1-Fluorenes		D	0.73	13.99	0.41
C2-Fluorenes		D	0.59	5.30	0.16
C3-Fluorenes		D	0.35	1.92	0.06
Phenanthrene	E	E	1.00	19.13	0.56
Anthracene		E	1.00	20.72	0.61
C1-Phenanthrenes/Anthracenes		E	0.57	7.44	0.22
C2-Phenanthrenes/Anthracenes		E	0.32	3.20	0.09
C3-Phenanthrenes/Anthracenes		E	0.29	1.26	0.04
C4-Phenanthrenes/Anthracenes		E	0.12	0.56	0.02
Fluoranthene	F	F	1.00	7.11	0.21
Pyrene	G	G	1.00	10.11	0.30
C1-Fluoranthenes/Pyrenes		G	0.51	4.89	0.14
Benz[a]anthracene		H	1.00	2.23	0.066
Chrysene	H	H	1.00	2.04	0.060
C1-Chrysenes/Benz[a]anthracenes		H	0.62	0.86	0.025

^A From Hawthorne, S. B., Grabanski, C.B., Miller, D. J., and Kreitinger, J. P., "Solid Phase Microextraction Measurement of Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," *Environmental Science Technology*, 39, 2005, pp. 2795-2803.

TABLE 3 Primary Material Hazards

Material	Hazards	Exposure Limit ^A	Signs and Symptoms of Exposure
Alum (Aluminum Potassium Sulfate)	Irritant	2 mg/M ³ TWA	May cause skin irritation, especially under repeated or prolonged contact, or when moisture is present. May irritate or burn the eyes. Dust or mist inhalation at levels above the TLV may cause irritation to the respiratory tract. May irritate the gastrointestinal tract.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Dichloromethane (DCM)	Carcinogen, Irritant	25 ppm-TWA, 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 mg/M ³ TWA	Causes skin irritation, chemical burns, permanent injury or scarring, and blindness. Vinegar is a mild acid that will neutralize lye if it were to make contact with the skin. Harmful if inhaled or ingested. Causes Sore throat, cough labored breathing, shortness of breath, and abdominal pain. Symptoms may be delayed.

^A Exposure limit refers to the OSHA regulatory exposure limit.

TABLE 4 SIM Ion Groups and Retention Time Windows

NOTE—Retention times must be verified by the user.

Analyte	SIM Ion Group	Target m/z	Retention Time (min)	
			Start	Stop
Naphthalene	1	128.1	7	17
2-Methylnaphthalene	1	142.1	7	17
1-Methylnaphthalene	1	142.1	7	17
C2-Naphthalenes	1	156.1	7	17
C3-Naphthalenes	1	170.1	7	17
C4-Naphthalenes	1,2	184.1	7	21
Acenaphthylene	1	152.1	7	17
Acenaphthene	1	154.1	7	17
Fluorene	1	166.1	7	17
C1-Fluorenes	2	180.1	17	21
C2-Fluorenes	2	194.1	17	21
C3-Fluorenes	2,3	208.1	17	25
Phenanthrene	2	178.1	17	21
Anthracene	2	178.1	17	21
C1-Phenanthrenes/Anthracenes	2	192.1	17	21
C2-Phenanthrenes/Anthracenes	2,3	206.1	17	30
C3-Phenanthrenes/Anthracenes	2,3	220.1	17	30
C4-Phenanthrenes/Anthracenes	3	234.1	21	30
Fluoranthene	2,3	202.1	17	30
Pyrene	2,3	202.1	17	30
C1-Fluoranthenes/pyrenes	3	216.1	21	30
Benz[a]anthracene	3	228.1	21	30
Chrysene	3	228.1	21	30
C1-Chrysenes	3	242.1	21	30
d-PAH Internal Standards				
Naphthalene-d8	1	136.1	7	17
1-Methylnaphthalene-d10	1	152.1	7	17
Acenaphthene-d10	1	164.1	7	17
Fluorene-d10	1	176.1	7	17
Phenanthrene-d10	2	188.1	17	21
Fluoranthene-d10	2,3	212.1	17	30
Pyrene-d10	2,3	212.1	17	30
Chrysene-d12	3	240.2	21	30

TABLE 5 Initial Calibration Standard Series

Analyte	DCM Stock Conc. mg/mL	LCL			UCL
		CS1	CS2	CS3	CS4
		ng/1.5 mL	ng/1.5 mL	ng/1.5 mL	ng/1.5 mL
Naphthalene	41.5	8.3	83	415	1660
1-Methylnaphthalene	23.9	4.78	47.8	239	956
2-Methylnaphthalene	20.42	4.084	40.84	204.2	816.8
Acenaphthylene	9.02	1.804	18.04	90.2	360.8
Acenaphthene	11	2.2	22	110	440
Fluorene	7.55	1.51	15.1	75.5	302
Anthracene	0.6	0.12	1.2	6	24
Phenanthrene	5.5	1.1	11	55	220
Fluoranthene	2.11	0.422	4.22	21.1	84.4
Pyrene	1.8	0.36	3.6	18	72
Benz[a]anthracene	0.08	0.016	0.16	0.8	3.2
Chrysene	0.03	0.006	0.06	0.3	1.2
Deuterated Analogs of Mix A Compounds	Stock Solution	CS1	CS2	CS3	CS4
Naphthalene-d8	5	50.0	50.0	50.0	50.0
1-Methylnaphthalene-d10	6	60.0	60.0	60.0	60.0
Acenaphthene-d10	1.23	12.3	12.3	12.3	12.3
Fluorene-d10	1.2	12.0	12.0	12.0	12.0
Phenanthrene-d10	0.96	9.6	9.6	9.6	9.6
Fluoranthene-d10	0.93	9.3	9.3	9.3	9.3
Pyrene-d10	0.84	8.4	8.4	8.4	8.4
Chrysene-d12	0.033	0.33	0.33	0.33	0.33

TABLE 6 Example of a 24-h Analytical Sequence^A

Example Analytical Sequence						
Run Type	Minutes	Cumulative Minutes to Start	Cumulative Minutes to End	Cumulative Hours to Start ^A	Cumulative Hours to End	
Standard	50	0	50	0.0	0.8	
Standard	50	50	100	0.8	1.7	
Blank	50	100	150	1.7	2.5	
Blank	50	150	200	2.5	3.3	
Sample	50	200	250	3.3	4.2	
Sample	50	250	300	4.2	5.0	
Blank	50	300	350	5.0	5.8	
Blank	50	350	400	5.8	6.7	
Sample	50	400	450	6.7	7.5	
Sample	50	450	500	7.5	8.3	
Blank	50	500	550	8.3	9.2	
Blank	50	550	600	9.2	10.0	
Sample	50	600	650	10.0	10.8	
Sample	50	650	700	10.8	11.7	
Blank	50	700	750	11.7	12.5	
Blank	50	750	800	12.5	13.3	
Sample	50	800	850	13.3	14.2	
Sample	50	850	900	14.2	15.0	
Blank	50	900	950	15.0	15.8	
Blank	50	950	1000	15.8	16.7	
Sample	50	1000	1050	16.7	17.5	
Sample	50	1050	1100	17.5	18.3	
Blank	50	1100	1150	18.3	19.2	

^A The last pore water sample must be injected within 24 h of the flocculation step (that is, the value for cumulative hours to start must be ≤ 24).

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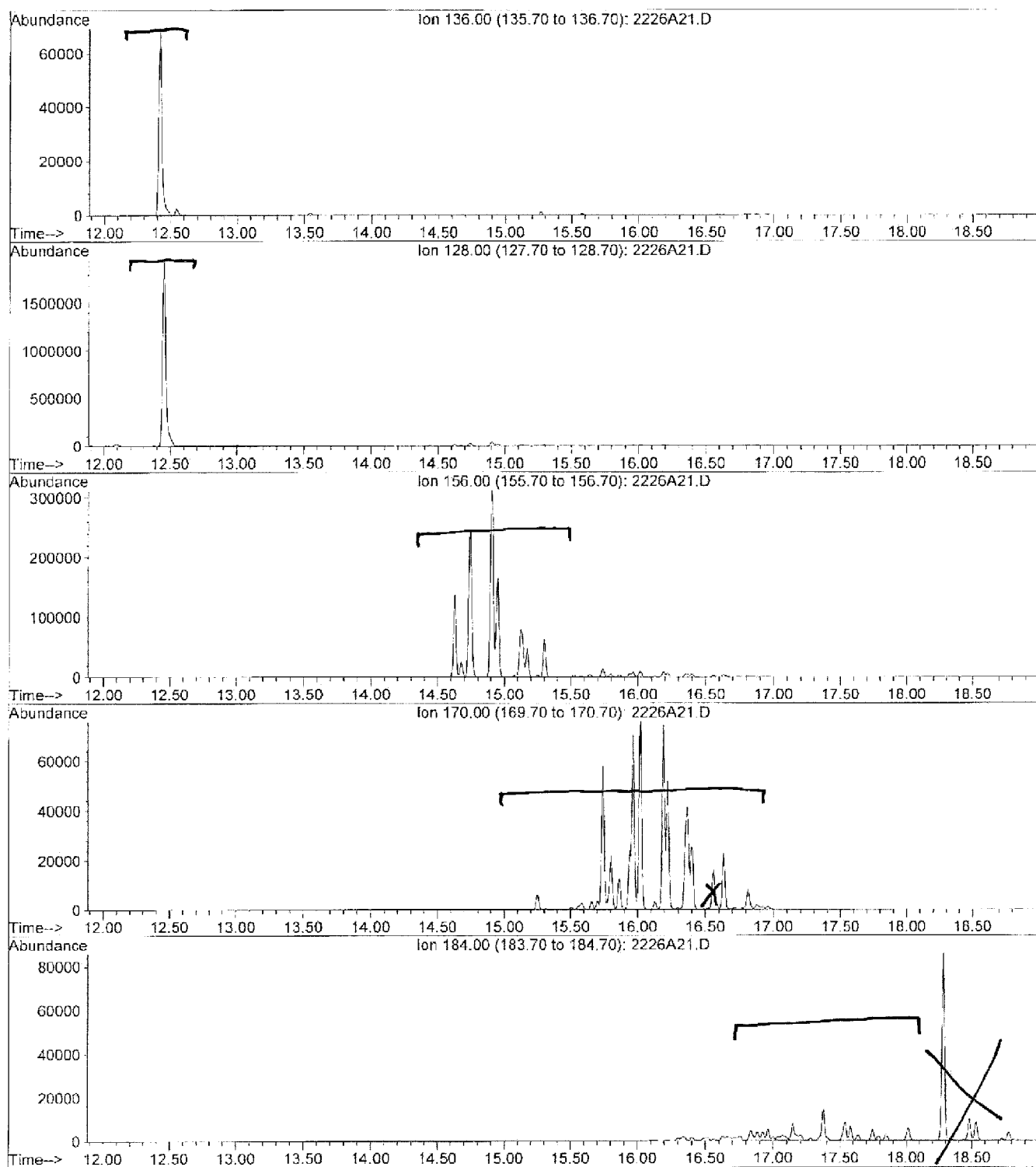


FIG. X1.1 Naphthalenes

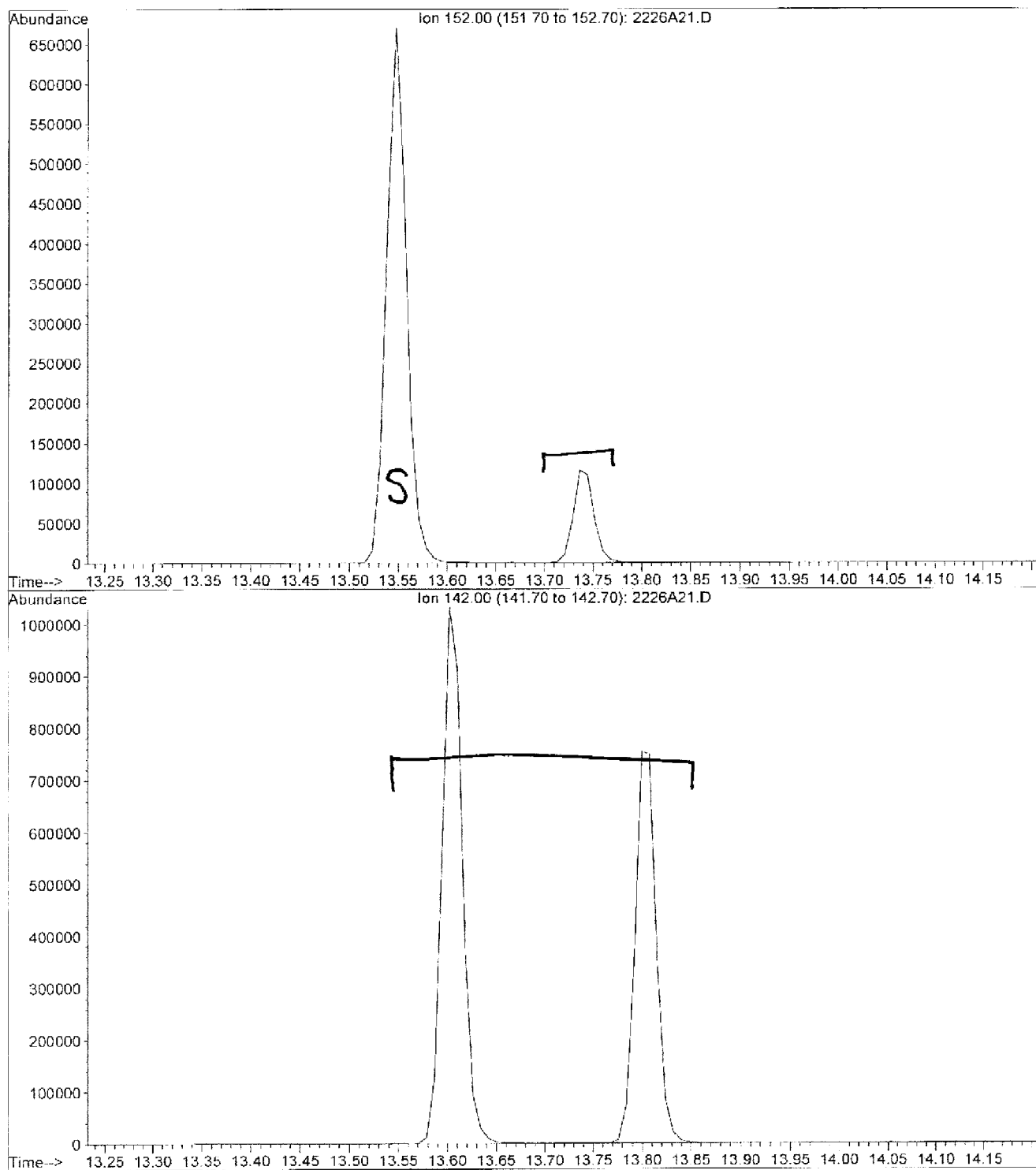


FIG. X1.2 Methyl naphthalenes
("s" is a spiked d₁₀-methyl naphthalene surrogate)

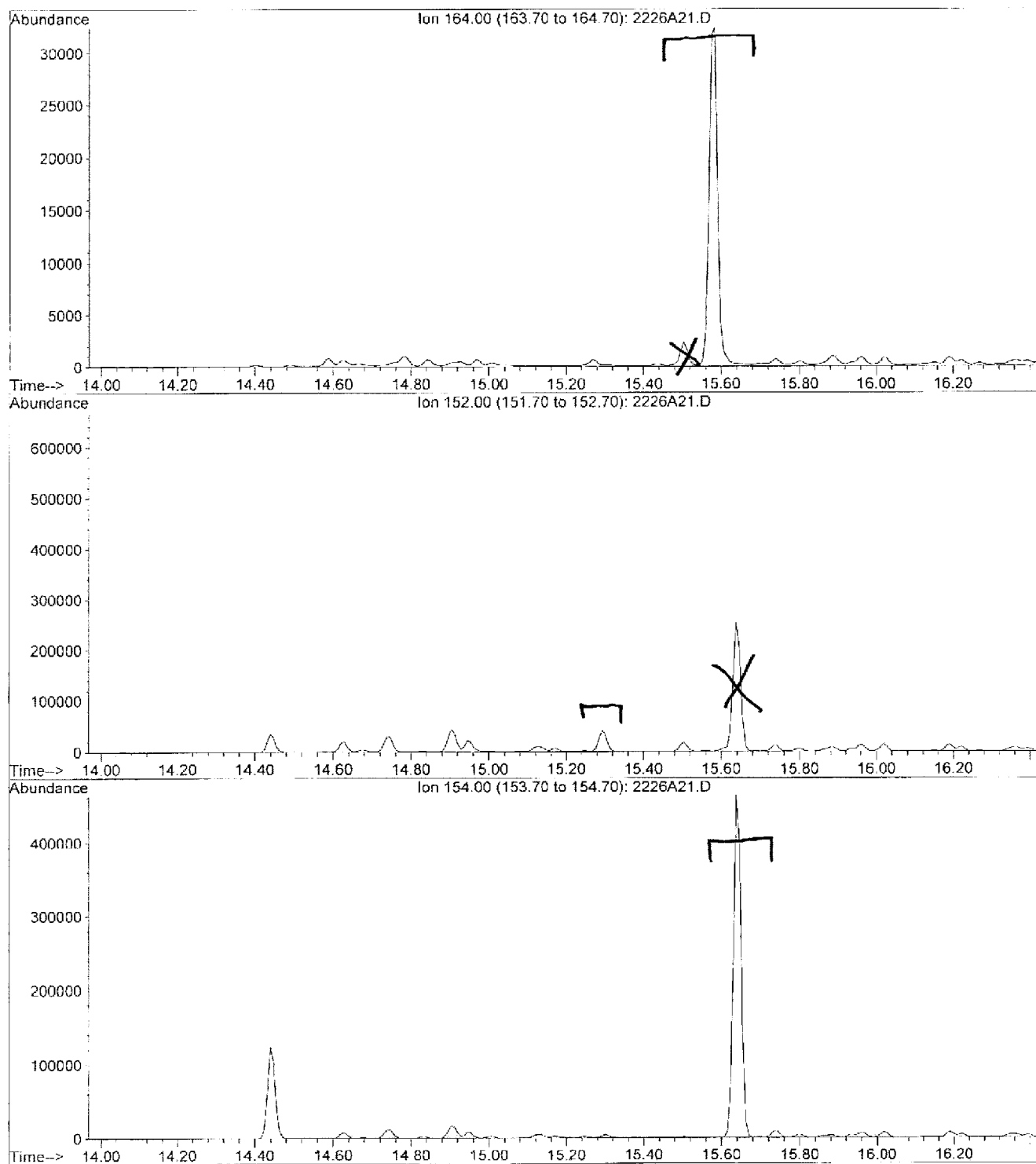


FIG. X1.3 Acenaphthylene/Acenaphthene

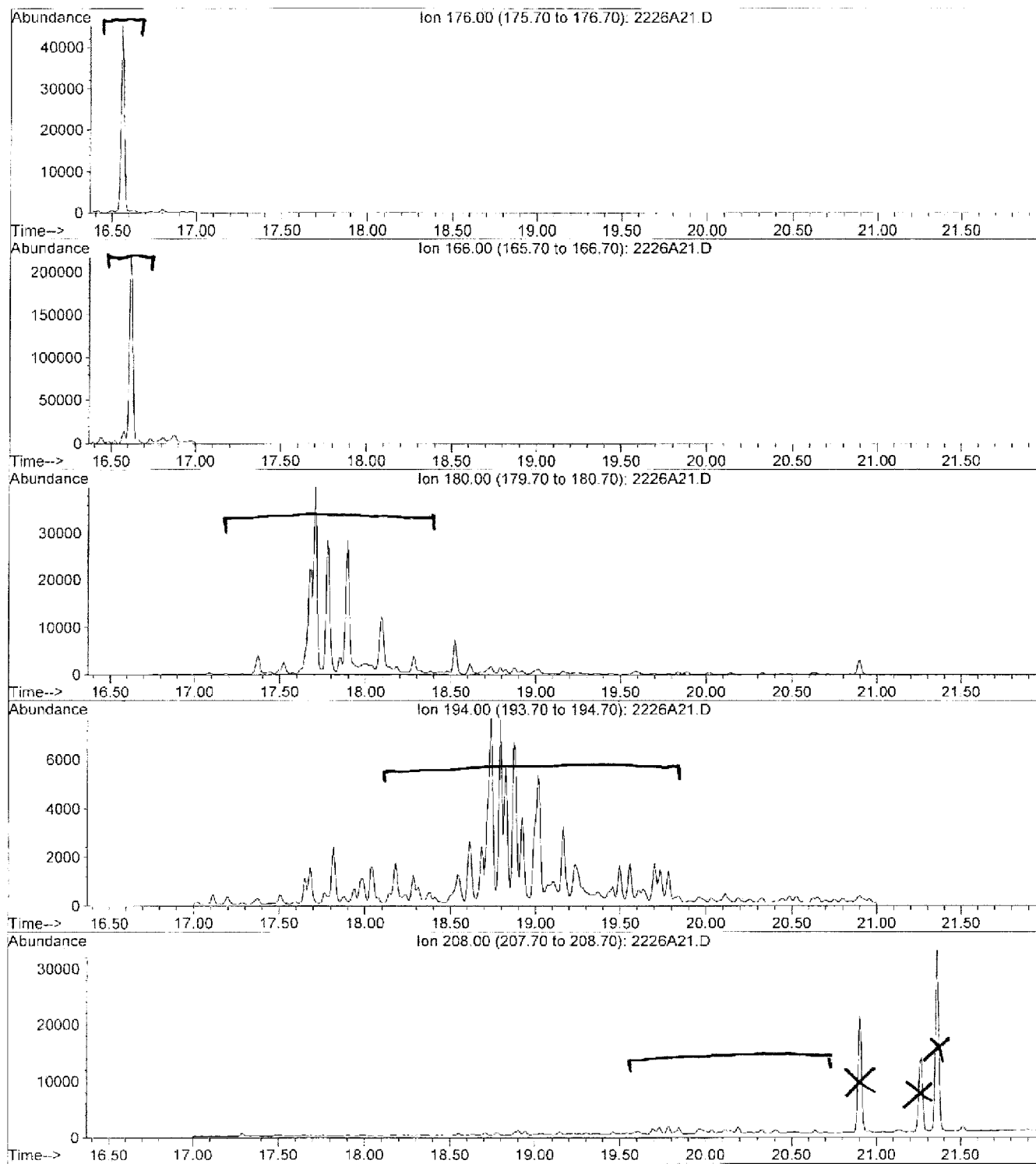


FIG. X1.4 Fluorenes

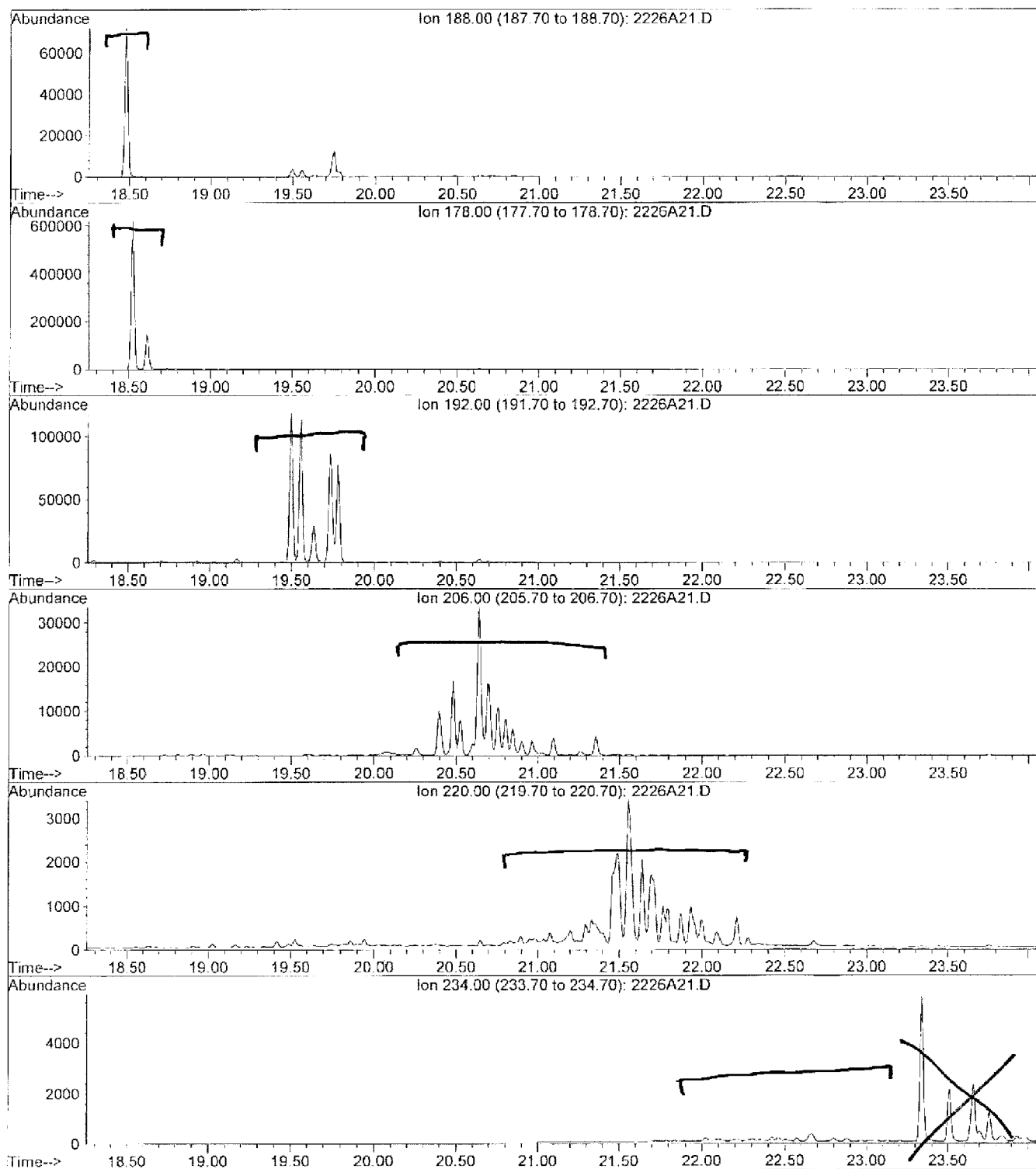


FIG. X1.5 Phenanthrenes/Anthracenes

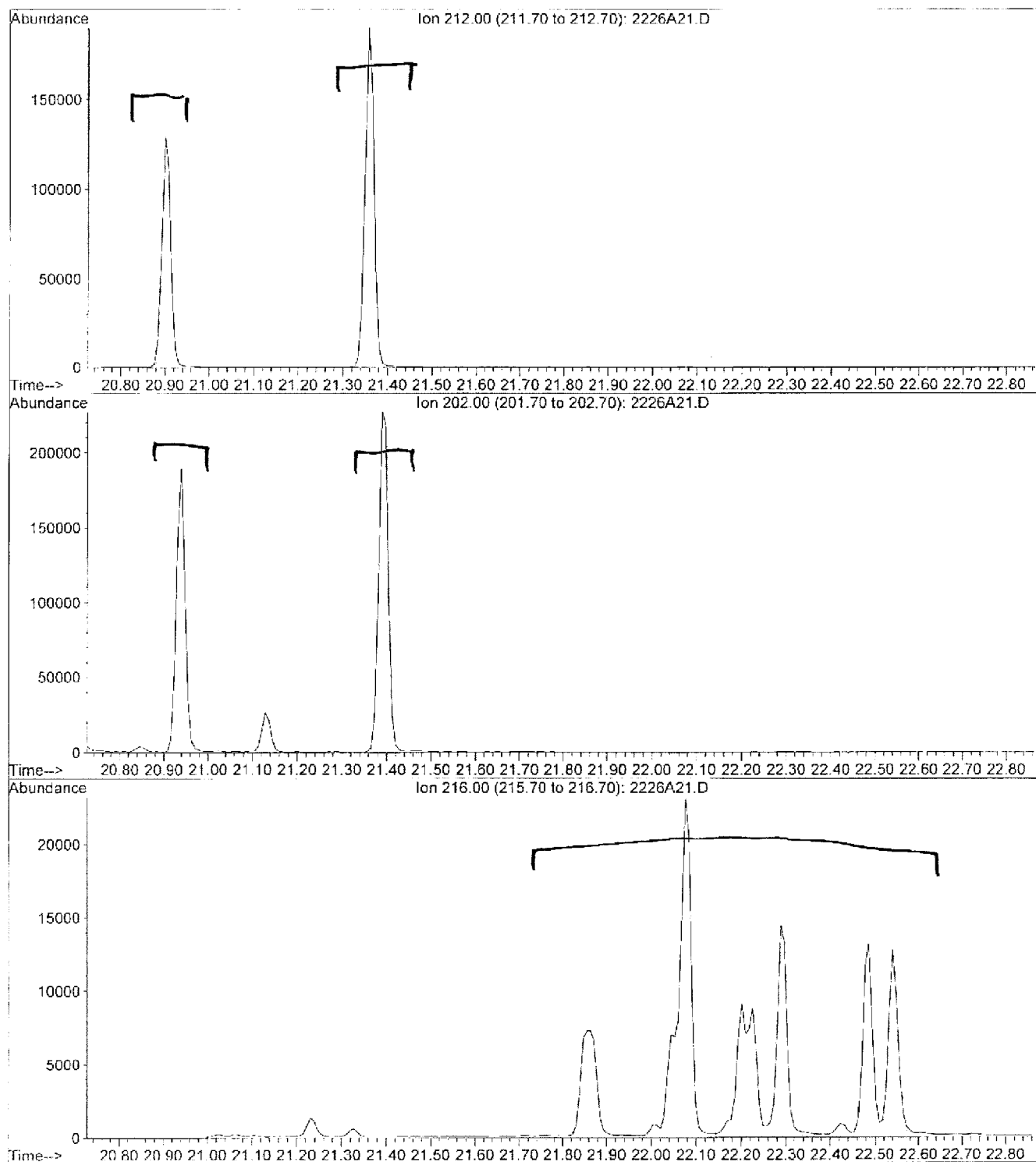


FIG. X1.6 Fluoranthenes/Pyrenes

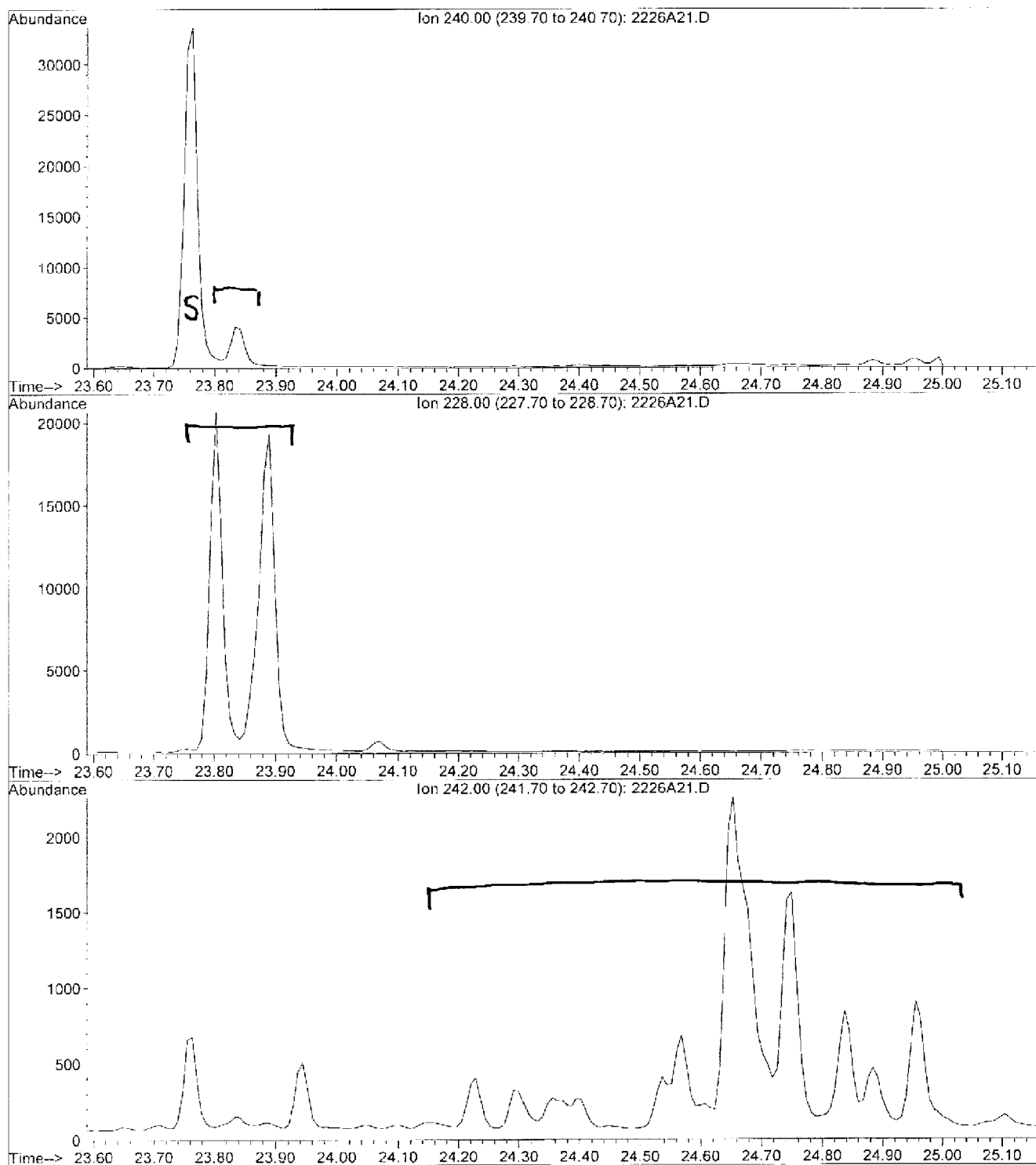


FIG. X1.7 Benz[a]anthracenes/Chrysenes
("s" is a spiked d₁₂-benz[a]anthracene surrogate)



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5

77 WEST JACKSON BOULEVARD

CHICAGO, IL 60604-3590

June 6, 2012

Mr. Narendra Prasad
Integrys Business Support, LLC
130 East Randolph Drive, 22nd Floor
Chicago, IL 60601

REPLY TO THE ATTENTION OF:

Subject: Review of the *Step II Sediment Sampling Work Plan, Revision 2, Former Wisconsin Public Service Corporation's Manitowoc Manufactured Gas Plant Site, Manitowoc, Wisconsin*, prepared for Integrys Business Support LLC, prepared by Natural Resource Technology, Inc. (dated April 30, 2012).

Dear Mr. Prasad:

US EPA reviewed the *Step II Sediment Sampling Work Plan, Revision 2, Former Wisconsin Public Service Corporation's Manitowoc Manufactured Gas Plant Site, Manitowoc, Wisconsin*, prepared by Natural Resource Technology, Inc. (April 30, 2012).

All comments have been addressed and incorporated into the *Step II Sediment Sampling Work Plan, Revision 2*. US EPA approves this document.

Thank you.

If you have any questions, please call me at (312) 886-6244.

Sincerely,

A handwritten signature in dark ink, appearing to read "Margaret Gielniewski".

Margaret Gielniewski
Remedial Project Manager
U.S. Environmental Protection Agency, Region 5
77 West Jackson Boulevard, SR-6J
Chicago, IL 60604-3507

CC: John Tielsch, U.S. EPA
Annette Weissbach, WDNR

BCC: Catherine Schripsema, CH2M Hill



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5

77 WEST JACKSON BOULEVARD

CHICAGO, IL 60604-3590

June 6, 2012

Mr. Narendra Prasad
Integrys Business Support, LLC
130 East Randolph Drive, 22nd Floor
Chicago, IL 60601

REPLY TO THE ATTENTION OF:

Subject: Review of the *Step II Sediment Sampling Work Plan, Revision 2, Former Wisconsin Public Service Corporation's Manitowoc Manufactured Gas Plant Site, Manitowoc, Wisconsin*, prepared for Integrys Business Support LLC, prepared by Natural Resource Technology, Inc. (dated April 30, 2012).

Dear Mr. Prasad:

US EPA reviewed the *Step II Sediment Sampling Work Plan, Revision 2, Former Wisconsin Public Service Corporation's Manitowoc Manufactured Gas Plant Site, Manitowoc, Wisconsin*, prepared by Natural Resource Technology, Inc. (April 30, 2012).

All comments have been addressed and incorporated into the *Step II Sediment Sampling Work Plan, Revision 2*. US EPA approves this document.

Thank you.

If you have any questions, please call me at (312) 886-6244.

Sincerely,

A handwritten signature in dark ink, appearing to read "Margaret Gielniewski".

Margaret Gielniewski
Remedial Project Manager
U.S. Environmental Protection Agency, Region 5
77 West Jackson Boulevard, SR-6J
Chicago, IL 60604-3507

CC: John Tielsch, U.S. EPA
Annette Weissbach, WDNR

BCC: Catherine Schripsema, CH2M Hill



Enclosure F

**Email from Bob Paulson
(WBS) to Margaret
Gielniewski (USEPA)
(August 3, 2017)**

From: [Paulson.Robert](#)
To: ["Margaret Gielniewski"](#)
Subject: FW: USEPA notification of SPME Sediment Sample Analysis
Date: Thursday, August 3, 2017 10:33:05 PM
Attachments: [EERC QA-QC, 9-16.pdf](#)
[Generic lab report, 8-2014.pdf](#)

Hello Margaret,

Please see below and attached for a slight change to the Two Rivers sampling next week.

Bob

-----Original Message-----

From: Brian Hennings [Brian.Hennings@obg.com]
Sent: Thursday, August 03, 2017 12:45 PM Central Standard Time
To: Paulson.Robert; Ken Mika
Cc: Jennifer Hagen
Subject: USEPA notification of SPME Sediment Sample Analysis

Hi Bob,

You can notify Margaret the following:

The laboratory that was approved by USEPA for SPME and bulk 34-PAH testing (META Environmental, Inc.) at the Manitowoc MGP site is not available to complete these analyses during this phase of sediment sampling at Two Rivers. In lieu of using META for this analysis, we will submit the samples to Steven Hawthorne at the Energy and Environmental Research Center (EERC) in North Dakota. Steve is a leader in solid phase micro extraction and measurement of PAHs for sediment risk assessment. He also assisted us with the selection of META for previous testing at Manitowoc. We have attached EERC's lab Quality Assurance procedures and a generic lab report for your review.

Let us know if you have any questions, or if you would like us to send her the notification. Thanks,
Brian



Brian G. Hennings, PG

NRT, an OBG Company | Senior Hydrogeologist
414-837-3524 | c 262-719-4512

Brian.Hennings@obg.com | www.obg.com/nrt

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From: Paulson.Robert [mailto:Robert.Paulson@we-energies.com]
Sent: Wednesday, August 02, 2017 3:09 PM
To: Ken Mika <Ken.Mika@obg.com>
Cc: Jennifer Hagen <Jennifer.Hagen@obg.com>; Brian Hennings <Brian.Hennings@obg.com>
Subject: RE: Two Rivers: SPME Sediment Samples

Proceed with getting EERC on board to do these and just run the contract through NRT. Also, prepare any notification to EPA and FSP/QAPP revisions necessary for the switch in labs.

Bob

From: Ken Mika [mailto:Ken.Mika@obg.com]
Sent: Wednesday, August 02, 2017 9:59 AM
To: Paulson.Robert
Cc: Jennifer Hagen; Brian Hennings
Subject: RE: Two Rivers: SPME Sediment Samples

Hi Bob,

The last time SPME and PAH-34 samples were collected was 2012, Manitowoc. The cost at the time for Manitowoc was \$12,300. In comparison to the this round, the cost is \$5,450 higher.

Ken

Kenneth R. Mika, PE

NRT, an OBG Company | Environmental Engineer
414-837-3572 | c 414-731-3111
Ken.Mika@obg.com | www.obg.com/nrt

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From: Paulson.Robert [mailto:Robert.Paulson@we-energies.com]
Sent: Tuesday, August 01, 2017 2:23 PM
To: Ken Mika <Ken.Mika@obg.com>
Cc: Jennifer Hagen <Jennifer.Hagen@obg.com>; Brian Hennings <Brian.Hennings@obg.com>
Subject: RE: Two Rivers: SPME Sediment Samples

Ken, what is the cost differential from what you had anticipated with switching to EERC?

Best Regards,
Bob

From: Ken Mika [mailto:Ken.Mika@obg.com]
Sent: Friday, July 28, 2017 2:25 PM
To: Paulson.Robert
Cc: Jennifer Hagen; Brian Hennings
Subject: Two Rivers: SPME Sediment Samples

Hi Bob,

Brian Hennings contacted Dave Mauro at META Environmental regarding SPME testing and was informed that their lab is no longer running the method. They are in the process of setting up a new lab, but it will not be ready in time for our samples. Dave was very helpful and did some calling around looking for alternative labs that can do the work reliably (which is really limited to META, Alpha, and the EERC in North Dakota). Alpha's equipment is off-line for repairs for the next 7 weeks, but North Dakota has availability (see cost below).

The cost for up to 10 porewater samples by ASTM D7363-13 (including the high MW PAHs) and up to 15 bulk sediment PAH-34 will be \$17,750. Any additional samples that arrive at the same time will cost \$840 for porewater analyses, and \$400 for bulk sediment PAH-34. Each porewater sample will include duplicate analyses and an associated blank (total of 3 GC/MS runs per sample) as required by the ASTM method. Bulk sediment analyses will be based on an 18-hour Soxhlet extraction. Please note that these prices are based on the reporting format and QA procedures outlined in the attached documents. As I am sure you can understand, any modifications of these formats or additional material (like raw peak areas etc) will add additional costs.

We have no technical concerns using the North Dakota lab, especially since Steve essentially wrote the method. But we should discuss USEPA notification and contracting.

Contracting:

I don't believe NRT or WBS have a contract with the North Dakota lab. META had a contract with IBS/WPSC and Dave told me that he would be willing to run things through his contract without markup. I think WBS would just have to update or create a new PO. Otherwise we can look into contracting North Dakota through WBS or NRT/OBG.

USEPA Notification:

META was approved to perform SPME in the Manitowoc SSWP after we attached a copy of the lab SOP. We are thinking that we could send Margaret an email with the attached EERC QA-QC document for their consideration and approval.

Let's talk as soon as you have a chance to read this over. Thanks,

Ken



Kenneth R. Mika, PE

NRT, an OBG Company | Environmental Engineer
234 W. Florida Street, Fifth Floor
Milwaukee, WI 53204
414-837-3572 | c 414-731-3111

Ken.Mika@obg.com | www.obg.com/nrt

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EERC Methods and Quality Assurance Procedures: 2016

EERC develops and performs research-grade analysis methods generally not available in contract laboratories. These procedures have all been vetted in the peer-reviewed literature, and some (e.g., the pore water PAH-34 method) are undergoing the studies required by certifying agencies (e.g., ASTM, EPA) as standard methods in preparation for their move to contract laboratories. Although some methods used are non-standard, EERC realizes that the resultant data may be used for regulatory purposes. Therefore, every effort is made to include method procedures and quality control procedures analogous to those used in standard contract lab analyses. Descriptions of the methods and quality assurance criteria are given below.

Laboratory Methods

Testing procedures are described in Table 1 and in the following peer-reviewed research literature:

- Total PAH-34 Measurements of Sediments as corrected for alkyl response factors as per [Hawthorne et al. 2006].

“Procedures for the Derivation of ESBs for the Protection of Benthic Organisms: PAH Mixtures,” EPA/600/R-02/013, Office of Research and Development, Washington D.C., 2003.

“Measurement of “Total” PAH Concentrations and Toxic Units Used for Sediment Risk Assessment at Manufactured Gas Plant Sites,” Hawthorne, Steven B., et al., 2005b, Energy & Environmental Research Center, University of North Dakota, Grand Forks, ND. Environmental Toxicology & Chemistry, Vol 25, No. 1, 2006, pp 287-296.

“Sampling and Analytical Methods of the National Status and Trends Program. Mussel Watch Project: 1993-1996,” U. S. Department of Commerce, 1998, Update, NOAA Technical Memorandum NOS ORCA 130, National Oceanic and Atmospheric Administration, Silver Spring, MD.

- Pore water PAH Determinations

“Solid-Phase Microextraction Measurement of Parent and Alkyl Polycyclic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values,” Hawthorne, Steven B., et al., Environmental Science & Technology, 39, 2005, pp 2795-2803.

ASTM Method D7363-13, "Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode." 2007.

“Measurement of Total PAH Concentrations and Toxic Units Used for Sediment Risk Assessment at Manufactured Gas Plant Sites,” Hawthorne, Steven B., et al., 2005b, Energy & Environmental Research Center, University of North Dakota, Grand Forks, ND. Environmental Toxicology & Chemistry, Vol. 25, No. 1, 2006, pp 287-296.

“Sampling and Analytical Methods of the National Status and Trends Program. Mussel Watch Project: 1993-1996,” U. S. Department of Commerce, 1998, Update, NOAA Technical Memorandum NOS ORCA 130, National Oceanic and Atmospheric Administration, Silver Spring, MD.

- Total Organic Carbon (TOC)

“Predicting Bioavailability of Sediment Polycyclic Aromatic Hydrocarbons to *Hyalella azteca* using Equilibrium Partitioning, Supercritical Fluid Extraction, and Pore Water Concentrations,” Hawthorne, Steven B., et al., Environmental Science & Technology, Vol. 41, No. 17, 2007, pp. 6297-6304.

- Soot Organic Carbon (SOC)

“Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability,” Gustafson, Orjan, et al., Environmental Science & Technology, Vol. 31, No. 1, 1997, pp. 203-209.

“Predicting Bioavailability of Sediment Polycyclic Aromatic Hydrocarbons to *Hyalella azteca* using Equilibrium Partitioning, Supercritical Fluid Extraction, and Pore Water Concentrations,” Hawthorne, Steven B., et al., Environmental Science & Technology, Vol. 41, No. 17, 2007, pp. 6297-6304.

- Dissolved Organic Carbon (DOC)

“Standard Methods for the Examination of Water and Wastewater,” EPA Method 5310C.

DOC values are determined after flocculation to remove colloidal carbon as described in:

“Solid-Phase Microextraction Measurement of Parent and Alkyl Polycyclic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values,” Hawthorne, Steven B., et al., Environmental Science & Technology, 39, 2005, pp 2795-2803.

ASTM Method D7363-13, "Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode." 2007.

DOC detection limits are: RL, 0.5 mg/L, MDL, 0.44 mg/L.

Pore Water and Sediment Detection Limits: PAH-34

Detection limits for each of the PAH-34 parent and groups of alkylated PAHs are given in Table 1. "Target" detection limits are based on the concentration of each PAH resulting in 1/34th of an EPA toxic unit [EPA, 2003]. However, actual detection limits routinely achieved by EERC are substantially lower in most cases (Table 1). In such cases, the measured values are reported as long as they meet the quality control guidelines discussed below.

Integration of Parent and Alkylated Clusters

Both the pore water and sediment PAH-34 methods result in chromatographic data that is much more complex than those encountered in standard laboratory methods. For example, more than 70 isomers of C4-alkyl naphthalenes are found in most samples, yet only account for one PAH on the PAH-34 list, and the PAH-34 list actually includes many hundreds of individual isomeric PAHs [Hawthorne et al. 2005, 2006] [ASTM D 7363]. Several interfering species must be eliminated from the integrated target peaks. All integrations must be monitored by an experienced analyst--typical automated integration algorithms are not sufficient [ASTM D 7363]. In many cases, hand integration by an experienced analyst is required. ASTM D7363 shows the chromatographic patterns of the target PAH-34 species in pore water along with the internal standards, and with non-target interfering species crossed out. Chromatographic patterns for sediment extracts are similar, except that the higher molecular weight PAHs have proportionately higher concentrations than in pore water.

Quality Control Criteria

Quality control criteria are summarized in Table 3. Formalized criteria for pore water analyses are those developed by EERC for ASTM method D 7363. Criteria for sediment PAH-34 analyses have not yet been approved by a methods agency, but are similar to the pore water criteria with the addition of surrogate recovery requirements.

All methods require initial 3- to 5-point standard curves meeting the criteria in Table 1 before any samples are analyzed. (The number of standard concentrations varies with PAH because of the varying detection limits and solubilities of the 2- to 6-ring PAHs.) After the multi-point standard calibration curve is achieved, duplicate daily calibrations are performed using a medium concentration standard that must meet the criteria stated in Table 3.

Duplicate blank water samples are analyzed before each pore water sample (which are required to be duplicates by ASTM D 7363), and the blank just previous to the sample analyses must meet the criteria stated in Table 3. For sediment Soxhlet extracts, solvent blanks are generated with each extraction set, and must meet the criteria stated in Table 3. Surrogate recovery requirements of 70 to 120% must be met, or no data is reported. In the rare cases where surrogate recovery (or solvent blank) criteria are not met, the entire extraction procedure is repeated on a fresh sample. As noted above, routine experimental detection limits achieved at EERC are normally lower than the

required "target" detection limits. Such data are reported as long as they meet the signal to noise, and blank requirements stated in Table 1. Values are flagged as "J" when they are below the lowest related calibration standard concentrations, and as "E" when the highest calibration standard is exceeded.

Samples are required to be shipped and stored at approximately 4 °C, and any deviations are noted on the COC forms. All PAH analyses are to be started within the 28 day holding time [ASTM D 7363]. TOC, SOC, and DOC analyses should begin within 40 days holding time.

Table 1: Laboratory Methods

PARAMETER	METHOD	METHOD REFERENCE(S)
Soil or Sediment PAH-34 (dry wt.)	18 hour Soxhlet extraction in 1:1 methylene chloride:acetone after mixing to dryness with Na ₂ SO ₄ . Solvent to sample ratio (mL/g) should be >50:1. Extracts analyzed by GC/MS using Selected Ion Monitoring (SIM) for measuring parent and alkylated PAHs	[Hawthorne et al., 2006], [NOAA, 1998]
PAH-34 –pore water	Centrifugation and flocculation with immediate addition of multiple d-PAH internal standards followed by solid phase microextraction and GC/MS analysis for measuring parent and alkylated PAHs	[Hawthorne et al., 2005], [ASTM, 2013]
Total Organic Carbon (TOC)	Sample acidified to remove carbonates, followed by analysis using a Leeman CE44 Elemental Analyzer modified for sediment analysis.	[Hawthorne et al., 2007]
Soot Organic Carbon (SOC)	Heat stable TOC following pretreatment at 375° C for 24 hrs. followed by analysis using a Leeman CE44 Elemental Analyzer modified for sediment analysis.	[Gustafson et al., 1997], [Hawthorne, 2007]
Dissolved Organic Carbon (DOC)	EPA method 5310C after flocculation.	[Standard Methods for the Examination of Water and Wastewater] [Hawthorne et al., 2005], [ASTM, 2013]

^aPAH-34 includes the 18 parent and 16 groups of alkylated PAHs specific in the U.S. EPA hydrocarbon narcosis model for PAHs [U.S. EPA, 2003], [Hawthorne et al., 2005], [NOAA, 1998], [Hawthorne et al., 2006]

Table 2: Pore Water and Sediment Detection Limits

PAHs	Method	Pore Water Target Detection Limits ¹ (ug/L)	Pore Water Actual Detection Limits ³ (ug/L)	Sediment Target Detection Limits ^{1,2} (mg/Kg dry wt)	Sediment Actual Detection Limits ³ (mg/Kg dry wt)
Naphthalene	GC/MS	5.7	0.500	0.11	0.001
2-Methylnaphthalene	GC/MS	2.4	0.200	0.13	0.001
1-Methylnaphthalene	GC/MS	2.4	0.200	0.13	0.001
C2 naphthalenes	GC/MS	0.89	0.600	0.15	0.005
C3 naphthalenes	GC/MS	0.33	0.300	0.17	0.010
C4 naphthalenes	GC/MS	0.12	0.050	0.19	0.010
Acenaphthylene	GC/MS	9.0	0.100	0.13	0.001
Acenaphthene	GC/MS	1.6	0.050	0.14	0.001
Fluorene	GC/MS	1.2	0.050	0.16	0.001
C1 fluorenes	GC/MS	0.41	0.100	0.18	0.005
C2 fluorenes	GC/MS	0.16	0.100	0.20	0.01
C3 fluorenes	GC/MS	0.06	0.050	0.23	0.030
Phenanthrene	GC/MS	0.56	0.200	0.18	0.001
Anthracene	GC/MS	0.61	0.050	0.17	0.001
C1 phenanthrenes/anthracenes	GC/MS	0.22	0.200	0.20	0.005
C2 phenanthrenes/anthracenes	GC/MS	0.09	0.050	0.22	0.010
C3 phenanthrenes/anthracenes	GC/MS	0.04	0.020	0.24	0.020
C4 phenanthrenes/anthracenes	GC/MS	0.02	0.020	0.27	0.030
Fluoranthene	GC/MS	0.21	0.040	0.21	0.001
Pyrene	GC/MS	0.30	0.040	0.21	0.001
C1 pyrene/fluoranthenes	GC/MS	0.14	0.050	0.23	0.005
Benz(a)anthracene	GC/MS	0.066	0.010	0.25	0.002
Chrysene	GC/MS	0.060	0.010	0.25	0.002
C1 benz(a)anthracene/chrysenes	GC/MS	0.025	0.020	0.27	0.010
C2 benz(a)anthracene/chrysenes	GC/MS	0.014	0.008	0.30	0.030
C3 benz(a)anthracene/chrysenes	GC/MS	0.005	0.008	0.33	0.050
C4 benz(a)anthracene/chrysenes	GC/MS	0.002	0.008	0.36	0.080
Benzo(b & k)fluoranthene ⁴	GC/MS	0.019	0.010	0.29	0.002
Benzo(a)pyrene	GC/MS	0.028	0.005	0.28	0.002
Benzo(e)pyrene	GC/MS	0.026	0.005	0.28	0.002
Perylene	GC/MS	0.026	0.005	0.28	0.002
Indeno(1,2,3-cd)pyrene	GC/MS	0.008	0.002	0.33	0.002
Dibenz(a,h)anthracene	GC/MS	0.008	0.002	0.33	0.002
Benzo(g,h,i)perylene	GC/MS	0.013	0.002	0.32	0.002

Table 2 Notes:

- (1) Target detection limits for both sediment and pore water are 1/34th of the concentration of each individual PAH that corresponds to one toxic unit as described in U. S. EPA (2003) *Equilibrium Partitioning Sediment Guidelines (ESGs) for the Protection of Benthic Organisms*.
- (2) Sediment target detection limits were estimated assuming 1.0 % total organic carbon.
- (3) Actual laboratory detection limit for sediments and pore water are based on previous research studies. Final reported detection limits are determined with each sample set and will vary based analyte levels and matrix interferences.
- (4) Benzo[b]fluoranthene and benzo[k]fluoranthene are reported as their sum because of insufficient chromatographic resolution.

Table 3: Quality Control Criteria

Quality Control Check	Sediment PAH-34 ^a	Pore Water PAH-34 [ASTM D7363-13]
GC/MS Tuning Criteria	As per manufacturer's instructions using PFTBA. Must pass criteria prior to analysis of each sample set or every 24 hrs.	As per manufacturer's instructions using PFTBA. Must pass criteria prior to analysis of each sample set or every 24 hrs.
Initial Calibration	Four or five-point initial calibration established with each sample set. The RSD (area ratio per ng) must be less than 30% for high molecular weight PAHs and less than 25% for low molecular weight PAHs, and the $r^2 > 0.99$. The calibration curve must not be forced through the origin.	Three- or four-point initial calibration established with each sample set. The RSD (area ratio per ng) must be less than 30% for high molecular weight PAHs and less than 25% for low molecular weight PAHs, and the $r^2 > 0.99$. The calibration curve must not be forced through the origin.
Daily Calibrations	Daily duplicate calibrations with a medium concentration standard. The mean area ratio per ng for the daily calibrations must agree with the mean area ratio per ng for the initial calibration curve within <30% for high molecular weight PAHs and <25% for low molecular weight PAHs.	Replicate calibration standards are analyzed at a medium level with each sample set. The mean area ratio per ng for the daily calibrations must agree with the mean area ratio per ng for the initial calibration curve within <25% for high molecular weight PAHs and <20% for low molecular weight PAHs.
Signal to noise	>10 to 1 for internal standards, >3 to one for target PAHs	>10 to 1 for internal standards, >3 to one for target PAHs
Surrogate Recoveries	70 to 120%, or sample is re-extracted	NA (ASTM D 7363-13)
Multiple Analyses	Quadruplicate samples are suggested because of sediment heterogeneity.	Duplicate samples are analyzed as required by ASTM D 7363-13.
Method Preparation Blanks	A solvent blank is prepared with each set of sediment extractions. Should the PAH concentrations in the blank be greater than 20% of the sample concentrations, the sample set is rejected except in cases where the sample results are <1/3 the target detection limit in Table 2.	Duplicate pure water blanks are analyzed between every sample water to monitor the baseline. Each blank just prior to the samples must have each PAH concentration below 1/3 the target detection limit (Table 2). Alternatively, if the blank concentrations are found to be <20% of the related sample, the data can be accepted.
Remedial action for failed calibrations, blanks, and surrogate recoveries.	Initial and daily calibrations must meet criteria before sample data can be reported. Failure to meet blank criteria and surrogate recovery criteria requires the extraction of a fresh sample.	Initial and daily calibrations must meet criteria before sample data can be reported. Failure to meet blank criteria requires the preparation and analysis of a fresh sample.



Report of Laboratory Analysis

XXXXXX, 2013

Dr. Steven B. Hawthorne

Energy and Environmental Research Center, Campus Box 9018
University of North Dakota, Grand Forks, North Dakota 58201
701-777-5256

Narrative

XXXXXX sediment samples were received on XXXXXXXXXX. All samples were in good condition, and had measured temperatures of XX to XX °C, as noted on the attached chain of custody forms. Each sample was subjected to an initial analysis to estimate the “34” PAH concentrations. These data were provided to XXXXX on an informal basis, and were used to select XX sediments for the determination of pore water PAH-34 concentrations, total PAH-34 concentrations, total organic carbon, soot organic carbon, and dissolved organic carbon as described in the methods document.

All analyses for reported data met all QA/QC criteria listed in the *EERC Methods and Quality Assurance Procedures: 2012* and those stated in ASTM method D7363. Pore waters were all analyzed in duplicate as required by ASTM method D7363. (Note: The ASTM method focuses on the lower molecular weight PAHs up to the methyl chrysenes that account for the majority of PAH toxicity to benthic organisms, but was extended for these investigations to measure the additional higher molecular weight PAHs listed that complete the EPA-34 list.) Soxhlet extracts of the sediments were performed in XXXXXX. All extracts met the 70 to 120% recovery criteria and all initial calibration, daily calibration, solvent blank, and water blank criteria stated in the ASTM method and the *EERC Methods and Quality Assurance Procedures: 2012* were met.

Data qualifiers are listed with each determination, and include “J” (for values estimated below the lowest calibration concentrations, but meeting the signal to noise requirements stated in the ASTM method), “E” (for values exceeding the highest calibration concentration), and “R” (for values rejected on the basis of the reasons described in the footnotes listed at the bottom of the table).



15 North 23rd Street
Grand Forks, ND 58203 USA
Ph: (701) 777-5000
Fax: (701) 777-5181

CHAIN OF CUSTODY FORM

FORM LP-01
REV. 02/2004

Client / Project:

Contact Name:

Address / Location:

Phone/Fax No.:

EERC Contact:

Phone Number:

SAMPLE INFORMATION						
Sample Identification	Matrix	Date/time Sampled	Collected By	Preservation	Analyses Requested/Comments	Lab Number

				CUSTODIAL LOCATIONS							
Sampled by (signature):		Company:		Date	Time	Received by (signature):		Company:		Date	Time
Printed name:				Sealed:	Y / N	Printed name:				Sealed:	Y / N
Relinquished by (signature):		Company:		Date	Time	Received by (signature):		Company:		Date	Time
Printed name:				Sealed:	Y / N	Printed name:				Sealed:	Y / N
Relinquished by (signature):		Company:		Date	Time	Received by (signature):		Company:		Date	Time
Printed name:				Sealed:	Y / N	Printed name:				Sealed:	Y / N

NOTES	

Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

[illegible]

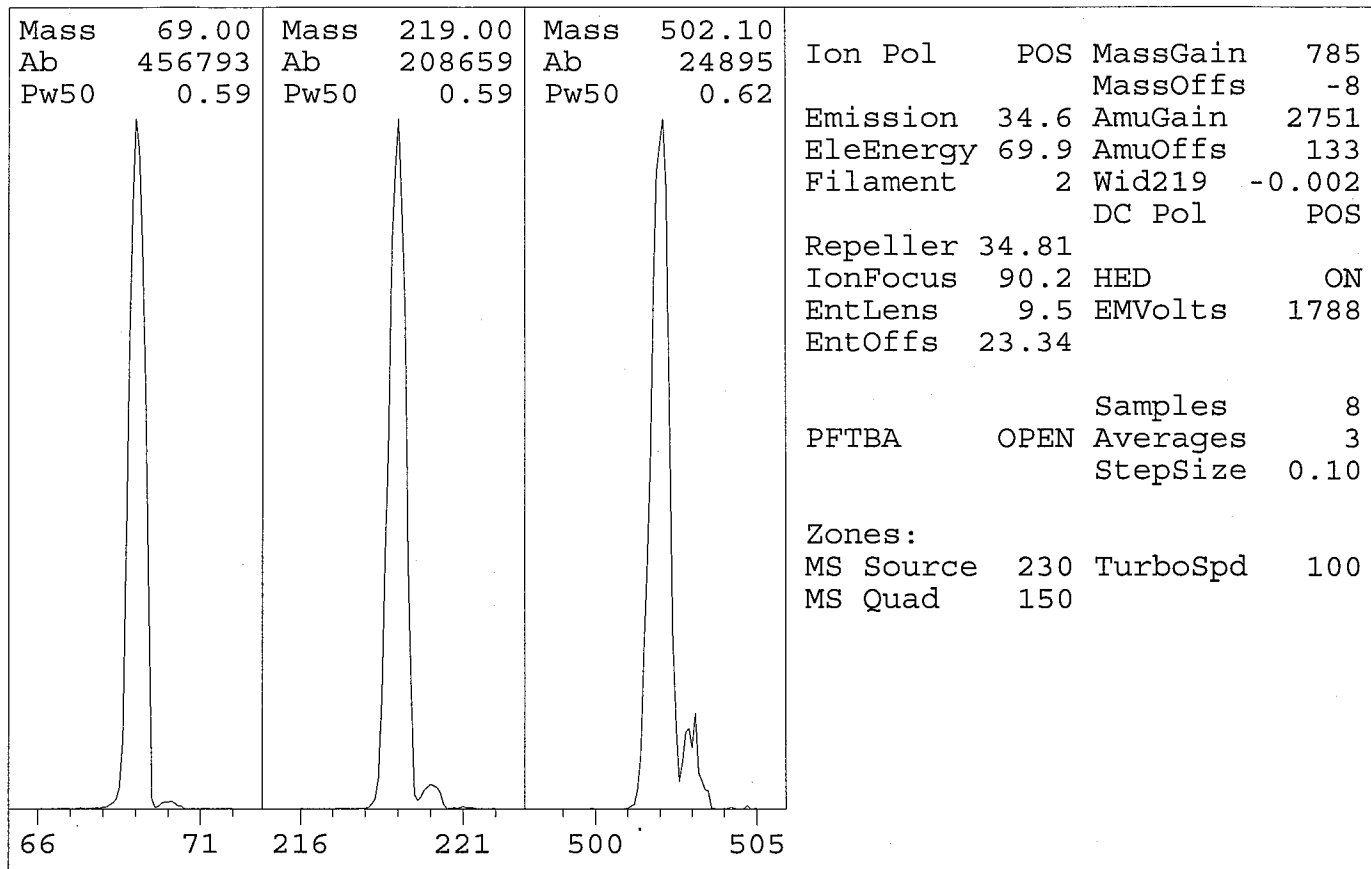
Energy and Environmental Research Center, GC/MS Lab
University of North Dakota, Campus Box 9018
15 North 23rd Street, Grand Forks, ND 58202
701-777-5000

[illegible]

Instrument: Instrument #1

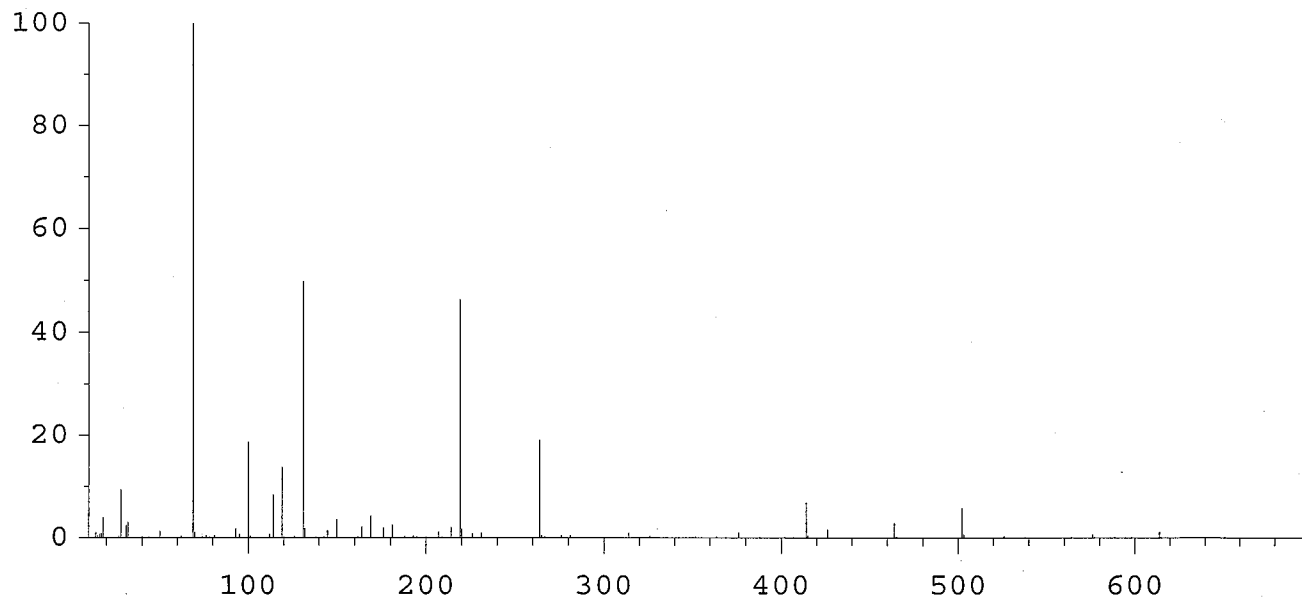
Thu May 29 08:10:18 2014

C:\MSDCHEM\1\5973N\ATUNE.U



Scan: 10.00 - 700.00 Samples: 8 Thresh: 100 Step: 0.10

276 peaks Base: 69.00 Abundance: 400576



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
69.00	400576	100.00	69.80	4468	1.12
219.00	185728	46.37	220.00	7219	3.89
502.00	23280	5.81	503.20	2714	11.66

Pore Water 4-point Calibration.

Analyte	lowest low medium high								ng in cal vial (1.5 mL)								Slope	Intercept	R2
	lowest	low	medium	high		lowest	low	medium	high		lowest	low	medium	high		lowest	low	medium	high
naphthalene	0.137	0.64	6.95	23.76			8.36	41.82	418.2	1672.8						0.014	0.31	0.9980	
2-methylnaphthalene	0.041	0.18	2.40	7.85			2.29	11.46	114.6	458.4						0.017	0.12	0.9963	
1-methylnaphthalene	0.036	0.17	2.14	7.10			2.49	12.44	124.4	497.6						0.014	0.10	0.9970	
C2 naphthalenes	0.14	0.64	6.95	23.76			8.36	41.82	418.2	1672.8						0.014	0.31	0.9980	
C3 naphthalenes	0.14	0.64	6.95	23.76			8.36	41.82	418.2	1672.8						0.014	0.31	0.9980	
C4 naphthalenes	0.14	0.64	6.95	23.76			8.36	41.82	418.2	1672.8						0.014	0.31	0.9980	
acenaphthylene	0.10	0.59	6.73	23.77			2.05	10.24	102.4	409.6						0.058	0.21	0.9987	
acenaphthene	0.15	0.74	8.52	28.07			2.20	10.98	109.8	439.2						0.063	0.44	0.9968	
fluorene	0.08	0.43	4.79	17.78			1.39	6.94	69.4	277.6						0.064	0.09	0.9996	
C1 fluorenes	0.08	0.43	4.79	17.78			1.39	6.94	69.4	277.6						0.064	0.09	0.9996	
C2 fluorenes	0.08	0.43	4.79	17.78			1.39	6.94	69.4	277.6						0.064	0.09	0.9996	
C3 fluorenes	0.08	0.43	4.79	17.78			1.39	6.94	69.4	277.6						0.064	0.09	0.9996	
phenanthrene	0.15	0.82	8.23	29.99			1.41	7.06	70.6	282.4						0.106	0.24	0.9994	
anthracene	0.02	0.10	1.16	5.11			0.22	1.12	11.2	45.0						0.114	-0.05	0.9995	
C1 phenanthrenes/anthracenes	0.15	0.82	8.23	29.99			1.41	7.06	70.6	282.4						0.106	0.24	0.9994	
C2 phenanthrenes/anthracenes	0.15	0.82	8.23	29.99			1.41	7.06	70.6	282.4						0.106	0.24	0.9994	
C3 phenanthrenes/anthracenes	0.15	0.82	8.23	29.99			1.41	7.06	70.6	282.4						0.106	0.24	0.9994	
C4 phenanthrenes/anthracenes	0.15	0.82	8.23	29.99			1.41	7.06	70.6	282.4						0.106	0.24	0.9994	
fluoranthene	0.05	0.30	3.17	12.57			0.49	2.43	24.3	97.4						0.129	0.00	1.0000	
pyrene	0.07	0.36	3.53	13.21			0.48	2.40	24.0	96.1						0.137	0.07	0.9997	
C1 fluoranthenes/pyrenes	0.07	0.36	3.53	13.21			0.48	2.40	24.0	96.1						0.137	0.07	0.9997	
benz[a]anthracene	0.39	0.87	2.54	3.62			0.16	0.41	1.63	2.45						1.393	0.23	0.9984	
chrysene	0.17	0.33	1.28	1.69			0.06	0.15	0.61	0.91						1.847	0.07	0.9925	
C1 chrysenes	0.39	0.87	2.54	3.62			0.06	0.15	0.61	0.91						3.751	0.23	0.9984	
C2 chrysenes	0.39	0.87	2.54	3.62			0.06	0.15	0.61	0.91						3.751	0.23	0.9984	
C3 chrysenes	0.39	0.87	2.54	3.62			0.06	0.15	0.61	0.91						3.751	0.23	0.9984	
C4 chrysenes	0.39	0.87	2.54	3.62			0.06	0.15	0.61	0.91						3.751	0.23	0.9984	
benzo[b+k]fluoranthene	0.14	0.41	1.51	2.04			0.06	0.14	0.56	0.85						2.419	0.05	0.9938	
benzo[e]pyrene	0.15	0.48	1.77	2.42			0.04	0.11	0.43	0.64						3.801	0.05	0.9948	
benzo[a] pyrene	0.10	0.25	1.03	1.51			0.06	0.14	0.56	0.84						1.810	0.00	0.9997	
perylene	0.03	0.09	0.35	0.49			0.02	0.05	0.19	0.29						1.697	0.01	0.9955	
indeno[1,2,3-cd]pyrene	0.01	0.03	0.10	0.15			0.01	0.02	0.08	0.12						1.208	0.00	0.9972	
dibenz[ah]anthracene	0.018	0.06	0.16	0.24			0.01	0.03	0.14	0.20						1.123	0.01	0.9967	
benzo[ghi]perylene	0.01	0.04	0.15	0.23			0.01	0.03	0.14	0.21						1.121	0.00	0.9997	

area ratio = target peak area divided by the area of the relevant internal standard

	area ratio per ng				4-point area ratios per ng			
	lowest	low	medium	high	Mean	SD	RSD	
naphthalene	0.0163	0.0152	0.0166	0.0142	0.016	0.001	7.1%	
2-methylnaphthalene	0.0179	0.0160	0.0210	0.0171	0.018	0.002	11.9%	
1-methylnaphthalene	0.0146	0.0135	0.0172	0.0143	0.015	0.002	10.7%	
C2 naphthalenes	0.0235	0.0219	0.0239	0.0205	0.022	0.002	7.1%	
C3 naphthalenes	0.0144	0.0134	0.0147	0.0125	0.014	0.001	7.1%	
C4 naphthalenes	0.0115	0.0107	0.0117	0.0100	0.011	0.001	7.1%	
acenaphthylene	0.0487	0.0575	0.0657	0.0580	0.057	0.007	12.1%	
acenaphthene	0.0663	0.0674	0.0776	0.0639	0.069	0.006	8.8%	
fluorene	0.0588	0.0622	0.0690	0.0641	0.064	0.004	6.7%	
C1 fluorenes	0.0431	0.0455	0.0505	0.0469	0.047	0.003	6.7%	
C2 fluorenes	0.0344	0.0364	0.0404	0.0375	0.037	0.003	6.7%	
C3 fluorenes	0.0207	0.0219	0.0243	0.0225	0.022	0.002	6.7%	
phenanthrene	0.1072	0.1163	0.1166	0.1062	0.112	0.006	5.1%	
anthracene	0.0764	0.0878	0.1031	0.1137	0.095	0.016	17.3%	
C1 phenanthrenes/anthracenes	0.0608	0.0659	0.0661	0.0602	0.063	0.003	5.1%	
C2 phenanthrenes/anthracenes	0.0346	0.0375	0.0376	0.0343	0.036	0.002	5.1%	
C3 phenanthrenes/anthracenes	0.0312	0.0338	0.0339	0.0309	0.032	0.002	5.1%	
C4 phenanthrenes/anthracenes	0.0125	0.0136	0.0136	0.0124	0.013	0.001	5.1%	
fluoranthene	0.1069	0.1221	0.1303	0.1291	0.122	0.011	8.8%	
pyrene	0.1363	0.1486	0.1469	0.1375	0.142	0.006	4.4%	
C1 fluoranthenes/pyrenes	0.0700	0.0763	0.0754	0.0706	0.073	0.003	4.4%	
benz[a]anthracene	2.4062	2.1296	1.5543	1.4781	1.892	0.450	23.8%	
chrysene	2.8183	2.1688	2.1158	1.8635	2.242	0.407	18.2%	
C1 chrysenes	4.0354	3.5715	2.6067	2.4789	3.173	0.754	23.8%	
C2 chrysenes	1.8564	1.6430	1.1991	1.1404	1.460	0.347	23.8%	
C3 chrysenes	1.4851	1.3144	0.9593	0.9123	1.168	0.277	23.8%	
C4 chrysenes	1.1138	0.9858	0.7195	0.6842	0.876	0.208	23.8%	
benzo[b+k]fluoranthene	2.4142	2.9324	2.6709	2.4073	2.606	0.250	9.6%	
benzo[e]pyrene	3.6110	4.5523	4.1475	3.7806	4.023	0.418	10.4%	
benzo[a] pyrene	1.8322	1.7696	1.8413	1.7993	1.811	0.033	1.8%	
perylene	1.5118	1.8961	1.8270	1.6749	1.727	0.171	9.9%	
indeno[1,2,3-cd]pyrene	1.2928	1.5231	1.1954	1.2462	1.314	0.145	11.0%	
dibenz[ah]anthracene	1.3597	1.6286	1.1872	1.1693	1.336	0.213	15.9%	
benzo[ghi]perylene	0.9901	1.1666	1.1041	1.1224	1.096	0.075	6.9%	

area ratio/ng = target peak area divided by the relevant internal standard area per ng of the target calibration compound

Pore Water Daily Calibrations

NOTE: Calibration dates match EERC analysis dates on each sample spreadsheet

	date		date		date		date		date		date		date		date	
	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng	daily cal area rat/ng	% deviation from mean 4-pt area rat/ng
naphthalene	0.016	2.4%	0.017	9.6%	0.017	9.6%	0.017	8.3%	0.017	8.3%	0.017	9.3%	0.017	9.3%	0.0168	7.6%
2-methylnaphthalene	0.018	1.9%	0.019	7.9%	0.019	7.0%	0.019	4.6%	0.019	4.7%	0.018	2.6%	0.018	2.1%	0.0182	1.1%
1-methylnaphthalene	0.015	1.9%	0.016	5.8%	0.016	5.7%	0.016	4.2%	0.016	5.0%	0.015	2.5%	0.015	1.7%	0.0149	0.1%
C2 naphthalenes	0.023	2.4%	0.025	9.6%	0.025	9.6%	0.024	8.3%	0.024	8.3%	0.025	9.3%	0.025	9.3%	0.0242	7.6%
C3 naphthalenes	0.014	2.4%	0.015	9.6%	0.015	9.6%	0.015	8.3%	0.015	8.3%	0.015	9.3%	0.015	9.3%	0.0148	7.6%
C4 naphthalenes	0.011	2.4%	0.012	9.6%	0.012	9.6%	0.012	8.3%	0.012	8.3%	0.012	9.3%	0.012	9.3%	0.0119	7.6%
acenaphthylene	0.061	5.7%	0.063	8.8%	0.059	2.2%	0.063	9.3%	0.061	5.4%	0.063	9.2%	0.061	6.7%	0.0613	6.6%
acenaphthene	0.068	1.6%	0.074	7.2%	0.074	7.4%	0.073	6.4%	0.073	5.8%	0.072	4.8%	0.075	9.4%	0.0723	5.2%
fluorene	0.058	8.5%	0.062	3.2%	0.063	0.2%	0.061	3.8%	0.062	2.9%	0.061	4.1%	0.062	1.9%	0.0609	4.2%
C1 fluorenes	0.043	8.5%	0.045	3.2%	0.046	0.2%	0.045	3.8%	0.045	2.9%	0.045	4.1%	0.046	1.9%	0.0446	4.2%
C2 fluorenes	0.034	8.5%	0.036	3.2%	0.037	0.2%	0.036	3.8%	0.036	2.9%	0.036	4.1%	0.037	1.9%	0.0356	4.2%
C3 fluorenes	0.020	8.5%	0.022	3.2%	0.022	0.2%	0.022	3.8%	0.022	2.9%	0.021	4.1%	0.022	1.9%	0.0214	4.2%
phenanthrene	0.101	9.1%	0.109	2.3%	0.112	0.8%	0.109	2.0%	0.114	1.7%	0.106	4.9%	0.105	5.8%	0.1043	6.5%
anthracene	0.089	6.2%	0.095	0.3%	0.093	2.5%	0.090	5.6%	0.103	8.1%	0.092	3.3%	0.088	7.4%	0.0915	3.9%
C1 phenanthrenes/anthracenes	0.058	9.1%	0.062	2.3%	0.064	0.8%	0.062	2.0%	0.064	1.7%	0.060	4.9%	0.060	5.8%	0.0592	6.5%
C2 phenanthrenes/anthracenes	0.033	9.1%	0.035	2.3%	0.036	0.8%	0.035	2.0%	0.037	1.7%	0.034	4.9%	0.034	5.8%	0.0337	6.5%
C3 phenanthrenes/anthracenes	0.030	9.1%	0.032	2.3%	0.033	0.8%	0.032	2.0%	0.033	1.7%	0.031	4.9%	0.031	5.8%	0.0303	6.5%
C4 phenanthrenes/anthracenes	0.012	9.1%	0.013	2.3%	0.013	0.8%	0.013	2.0%	0.013	1.7%	0.012	4.9%	0.012	5.8%	0.0122	6.5%
fluoranthene	0.113	7.5%	0.111	9.5%	0.117	3.8%	0.113	7.1%	0.117	3.9%	0.111	9.1%	0.113	7.2%	0.1101	9.8%
pyrene	0.133	6.7%	0.135	5.4%	0.132	7.2%	0.135	5.4%	0.129	9.2%	0.130	8.6%	0.129	9.6%	0.1310	8.0%
C1 fluoranthenes/pyrenes	0.068	6.7%	0.069	5.4%	0.068	7.2%	0.069	5.4%	0.066	9.2%	0.067	8.6%	0.066	9.6%	0.0672	8.0%
benz[a]anthracene	2.021	6.8%	2.003	5.9%	1.717	9.2%	2.034	7.5%	1.995	5.4%	2.024	7.0%	1.962	3.7%	2.0800	9.9%
chrysene	2.257	0.7%	2.427	8.3%	2.217	1.1%	2.364	5.5%	2.350	4.8%	2.267	1.1%	2.338	4.3%	2.4549	9.5%
C1 chrysenes	3.389	6.8%	3.359	5.9%	2.880	9.2%	3.411	7.5%	3.345	5.4%	3.395	7.0%	3.290	3.7%	3.4883	9.9%
C2 chrysenes	1.559	6.8%	1.545	5.9%	1.325	9.2%	1.569	7.5%	1.539	5.4%	1.562	7.0%	1.514	3.7%	1.6047	9.9%
C3 chrysenes	1.247	6.8%	1.236	5.9%	1.060	9.2%	1.255	7.5%	1.231	5.4%	1.249	7.0%	1.211	3.7%	1.2837	9.9%
C4 chrysenes	0.936	6.8%	0.927	5.9%	0.795	9.2%	0.941	7.5%	0.923	5.4%	0.937	7.0%	0.908	3.7%	0.9628	9.9%
benzo[b+k]fluoranthene	2.804	7.6%	2.787	6.9%	2.437	6.5%	2.707	3.8%	2.633	1.0%	2.414	7.4%	2.472	5.1%	2.6836	3.0%
benzo[e]pyrene	3.995	0.7%	4.157	3.3%	4.394	9.2%	4.339	7.9%	3.707	7.8%	4.090	1.7%	3.746	6.9%	3.7279	7.3%
benzo[a] pyrene	1.960	8.2%	1.859	2.6%	1.653	8.7%	1.827	0.9%	1.709	5.6%	1.972	8.9%	1.953	7.9%	1.9289	6.5%
perylene	1.864	7.9%	1.654	4.3%	1.604	7.2%	1.815	5.1%	1.623	6.1%	1.609	6.9%	1.732	0.3%	1.8606	7.7%
indeno[1,2,3-cd]pyrene	1.409	7.2%	1.433	9.0%	1.433	9.0%	1.433	9.1%	1.228	6.6%	1.428	8.6%	1.380	5.0%	1.2359	6.0%
dibenz[ah]anthracene	1.217	8.9%	1.240	7.2%	1.289	3.5%	1.394	4.3%	1.339	0.2%	1.328	0.6%	1.282	4.0%	1.2695	5.0%
benzo[ghi]perylene	1.133	3.4%	1.051	4.1%	1.177	7.4%	1.200	9.5%	1.150	4.9%	1.075	1.9%	1.185	8.1%	1.1280	2.9%

area rat/ng = target peak area divided by the relevant internal standard area per ng of the target calibration compound

Sediment 5-point Calibration

Analyte	area ratio					ug cal std					Slope	Intercept	R2
	uL Stock		uL 1:100 Stock			uL Stock		uL 1:100 Stock					
	100	10	100	50	10	100	10	100	50	10			
naphthalene	22.158	2.539	0.236	0.115	0.021	108.3	10.83	1.083	0.5415	0.1083	0.2041	0.081	0.9998
2-methylnaphthalene	14.352	1.541	0.140	0.068	0.014	47	4.7	0.47	0.235	0.047	0.3050	0.023	0.9999
1-methylnaphthalene	18.005	1.947	0.172	0.084	0.015	62.8	6.28	0.628	0.314	0.0628	0.2864	0.030	0.9999
C2 naphthalenes	4.436	0.570	0.052	0.027	0.005	61	6.1	0.61	0.305	0.061	0.0723	0.033	0.9992
C3 naphthalenes	2.161	0.217	0.020	0.010	0.002	27.8	2.78	0.278	0.139	0.0278	0.0778	0.000	1.0000
C4 naphthalenes	5.540	0.635	0.059	0.029	0.005	108.3	10.83	1.083	0.5415	0.1083	0.0510	0.020	0.9998
acenaphthylene	34.481	3.363	0.313	0.148	0.031	95.4	9.54	0.954	0.477	0.0954	0.3618	-0.036	1.0000
acenaphthene	21.341	2.343	0.205	0.099	0.018	88	8.8	0.88	0.44	0.088	0.2422	0.044	0.9999
fluorene	23.256	2.552	0.218	0.103	0.019	95.6	9.56	0.956	0.478	0.0956	0.2430	0.045	0.9999
C1 fluorenes	1.524	0.137	0.012	0.006	0.001	9.9	0.99	0.099	0.0495	0.0099	0.1544	-0.005	0.9999
C2 fluorenes	9.302	1.021	0.087	0.041	0.007	95.6	9.56	0.956	0.478	0.0956	0.0972	0.018	0.9999
C3 fluorenes	5.814	0.638	0.054	0.026	0.005	95.6	9.56	0.956	0.478	0.0956	0.0607	0.011	0.9999
phenanthrene	30.859	3.615	0.315	0.152	0.027	130.4	13.04	1.304	0.652	0.1304	0.2360	0.125	0.9997
anthracene	13.646	1.390	0.116	0.066	0.011	112.6	11.26	1.126	0.563	0.1126	0.1212	0.000	1.0000
C1 phenanthrenes/anthracenes	5.485	0.506	0.049	0.024	0.004	36.4	3.64	0.364	0.182	0.0364	0.1510	-0.013	0.9999
C2 phenanthrenes/anthracenes	0.322	0.029	0.003	0.001	0.000	11.6	1.16	0.116	0.058	0.0116	0.0279	-0.001	0.9999
C3 phenanthrenes/anthracenes	4.629	0.542	0.047	0.023	0.004	130.4	13.04	1.304	0.652	0.1304	0.0354	0.019	0.9997
C4 phenanthrenes/anthracenes	1.056	0.097	0.008	0.004	0.001	27.9	2.79	0.279	0.1395	0.0279	0.0379	-0.003	0.9999
fluoranthene	24.684	2.721	0.238	0.125	0.024	102.2	10.22	1.022	0.511	0.1022	0.2412	0.057	0.9999
pyrene	22.869	2.588	0.217	0.104	0.019	93.9	9.39	0.939	0.4695	0.0939	0.2431	0.064	0.9998
C1 fluoranthenes/pyrenes	1.581	0.137	0.015	0.007	0.002	10.1	1.01	0.101	0.0505	0.0101	0.1570	-0.006	0.9998
benz[a]anthracene	25.123	2.538	0.201	0.095	0.020	73.3	7.33	0.733	0.3665	0.0733	0.3430	-0.017	1.0000
chrysene	24.728	2.712	0.226	0.108	0.026	69	6.9	0.69	0.345	0.069	0.3580	0.047	0.9999
C1 chrysenes	11.622	1.274	0.106	0.051	0.012	69	6.9	0.69	0.345	0.069	0.1682	0.022	0.9999
C2 chrysenes	6.182	0.678	0.057	0.027	0.007	69	6.9	0.69	0.345	0.069	0.0895	0.012	0.9999
C3 chrysenes	4.946	0.542	0.045	0.022	0.005	69	6.9	0.69	0.345	0.069	0.0716	0.009	0.9999
C4 chrysenes	3.709	0.407	0.034	0.016	0.004	69	6.9	0.69	0.345	0.069	0.0537	0.007	0.9999
benzo[b+k]fluoranthene	85.940	9.455	0.738	0.403	0.084	80.7	8.07	0.807	0.4035	0.0807	1.0637	0.164	0.9999
benzo[e]pyrene	16.272	1.778	0.150	0.075	0.016	15.8	1.58	0.158	0.079	0.0158	1.0287	0.031	0.9999
benzo[a] pyrene	35.700	3.402	0.348	0.141	0.032	45.6	4.56	0.456	0.228	0.0456	0.7838	-0.053	1.0000
perylene	19.120	1.868	0.152	0.087	0.019	23.3	2.33	0.233	0.1165	0.0233	0.8215	-0.023	1.0000
indeno[1,2,3-cd]pyrene	9.510	0.738	0.084	0.039	0.008	7.9	0.79	0.079	0.0395	0.0079	1.2089	-0.056	0.9995
dibenz[ah]anthracene	31.515	2.720	0.219	0.125	0.027	19.4	1.94	0.194	0.097	0.0194	1.6300	-0.137	0.9998
benzo[ghi]perylene	35.267	3.399	0.276	0.134	0.026	20.6	2.06	0.206	0.103	0.0206	1.7148	-0.064	1.0000

area ratio = target peak area divided by the area of the relevant internal standard

	Area Ratio/ug					Mean	SD	%RSD
	high	med high	medium	low	lowest			
naphthalene	0.205	0.234	0.217	0.213	0.194	0.217	0.013	5.8%
2-methylnaphthalene	0.305	0.328	0.298	0.291	0.288	0.306	0.016	5.3%
1-methylnaphthalene	0.287	0.310	0.273	0.267	0.240	0.284	0.019	6.7%
C2 naphthalenes	0.073	0.093	0.086	0.088	0.082	0.085	0.009	10.3%
C3 naphthalenes	0.078	0.078	0.071	0.072	0.087	0.075	0.004	5.0%
C4 naphthalenes	0.051	0.059	0.054	0.053	0.048	0.054	0.003	5.8%
acenaphthylene	0.361	0.353	0.328	0.311	0.322	0.338	0.023	6.9%
acenaphthene	0.243	0.266	0.233	0.226	0.204	0.242	0.018	7.3%
fluorene	0.243	0.267	0.228	0.216	0.194	0.238	0.022	9.3%
C1 fluorenes	0.154	0.139	0.122	0.118	0.120	0.133	0.016	12.4%
C2 fluorenes	0.097	0.107	0.091	0.086	0.078	0.095	0.009	9.3%
C3 fluorenes	0.061	0.067	0.057	0.054	0.049	0.060	0.006	9.3%
phenanthrene	0.237	0.277	0.241	0.232	0.210	0.247	0.021	8.3%
anthracene	0.121	0.123	0.103	0.118	0.100	0.116	0.009	8.0%
C1 phenanthrenes/anthracenes	0.151	0.139	0.134	0.134	0.123	0.139	0.008	5.7%
C2 phenanthrenes/anthracenes	0.028	0.025	0.022	0.021	0.023	0.024	0.003	12.4%
C3 phenanthrenes/anthracenes	0.035	0.042	0.036	0.035	0.032	0.037	0.003	8.3%
C4 phenanthrenes/anthracenes	0.038	0.035	0.028	0.029	0.032	0.032	0.005	14.3%
fluoranthene	0.242	0.266	0.233	0.244	0.238	0.246	0.014	5.8%
pyrene	0.244	0.276	0.232	0.221	0.200	0.243	0.024	9.7%
C1 fluoranthenes/pyrenes	0.157	0.136	0.145	0.134	0.151	0.143	0.010	7.3%
benz[a]anthracene	0.343	0.346	0.275	0.259	0.271	0.306	0.045	14.7%
chrysene	0.358	0.393	0.328	0.314	0.382	0.348	0.035	10.0%
C1 chrysenes	0.168	0.185	0.154	0.148	0.180	0.164	0.016	10.0%
C2 chrysenes	0.090	0.098	0.082	0.079	0.096	0.087	0.009	10.0%
C3 chrysenes	0.072	0.079	0.066	0.063	0.076	0.070	0.007	10.0%
C4 chrysenes	0.054	0.059	0.049	0.047	0.057	0.052	0.005	10.0%
benzo[b+k]fluoranthene	1.065	1.172	0.915	0.998	1.041	1.037	0.109	10.5%
benzo[e]pyrene	1.030	1.126	0.952	0.951	1.014	1.014	0.083	8.2%
benzo[a] pyrene	0.783	0.746	0.764	0.617	0.710	0.728	0.075	10.3%
perylene	0.821	0.802	0.652	0.744	0.821	0.755	0.076	10.1%
indeno[1,2,3-cd]pyrene	1.204	0.935	1.060	0.988	0.951	1.047	0.117	11.1%
dibenz[ah]anthracene	1.624	1.402	1.130	1.289	1.382	1.362	0.208	15.3%
benzo[ghi]perylene	1.712	1.650	1.342	1.296	1.277	1.500	0.211	14.1%

area rat/ug = target peak area divided by the relevant internal standard area per ng of the target calibration compound

Sediment Daily Calibrations

NOTE: Calibration dates match EERC analysis dates on each sample spreadsheet

	date		date		date		date		date		date		date		date		date	
	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation	daily cal	% deviation
	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean	area rat/ug	from mean
	5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug		5-pt area rat/ug	
naphthalene	0.232	9.3%	0.217	2.1%	0.231	8.4%	0.226	6.2%	0.212	0.5%	0.229	7.5%	0.232	9.1%	0.219	3.0%	0.222	4.4%
2-methylnaphthalene	0.325	7.5%	0.328	8.5%	0.321	6.2%	0.328	8.6%	0.323	7.0%	0.319	5.8%	0.329	8.9%	0.303	0.3%	0.305	0.8%
1-methylnaphthalene	0.306	11.1%	0.293	6.5%	0.301	9.4%	0.294	6.7%	0.284	3.1%	0.294	6.8%	0.289	5.0%	0.291	5.8%	0.292	6.2%
C2 naphthalenes	0.097	15.5%	0.091	8.5%	0.092	8.8%	0.091	7.7%	0.090	7.3%	0.091	7.7%	0.091	8.4%	0.092	8.6%	0.092	9.6%
C3 naphthalenes	0.083	8.1%	0.080	3.5%	0.083	7.7%	0.082	5.9%	0.082	6.4%	0.081	5.4%	0.079	2.8%	0.076	1.6%	0.073	5.5%
C4 naphthalenes	0.058	9.3%	0.054	2.1%	0.058	8.4%	0.056	6.2%	0.053	0.5%	0.057	7.5%	0.058	9.1%	0.055	3.0%	0.055	4.4%
acenaphthylene	0.391	16.7%	0.367	9.4%	0.347	3.5%	0.357	6.6%	0.309	7.7%	0.316	5.6%	0.353	5.5%	0.364	8.6%	0.358	6.8%
acenaphthene	0.241	3.0%	0.243	3.9%	0.236	0.9%	0.239	2.1%	0.238	1.7%	0.232	1.0%	0.236	0.5%	0.223	4.8%	0.243	3.6%
fluorene	0.262	14.2%	0.243	5.8%	0.236	2.6%	0.231	0.6%	0.227	1.2%	0.234	1.8%	0.249	8.3%	0.242	5.5%	0.245	6.9%
C1 fluorenes	0.145	10.7%	0.135	3.0%	0.138	5.9%	0.132	1.0%	0.139	6.3%	0.134	2.6%	0.139	6.5%	0.143	9.3%	0.139	6.6%
C2 fluorenes	0.105	14.2%	0.097	5.8%	0.094	2.6%	0.092	0.6%	0.091	1.2%	0.094	1.8%	0.100	8.3%	0.097	5.5%	0.098	6.9%
C3 fluorenes	0.066	14.2%	0.061	5.8%	0.059	2.6%	0.058	0.6%	0.057	1.2%	0.058	1.8%	0.062	8.3%	0.061	5.5%	0.061	6.9%
phenanthrene	0.270	12.7%	0.247	3.0%	0.263	9.7%	0.245	2.0%	0.254	5.8%	0.262	9.2%	0.262	9.4%	0.251	4.7%	0.254	6.2%
anthracene	0.129	14.0%	0.109	3.4%	0.114	0.8%	0.111	1.5%	0.111	1.5%	0.122	8.0%	0.108	4.0%	0.121	7.2%	0.109	4.0%
C1 phenanthrenes/anthracenes	0.152	12.0%	0.146	7.1%	0.147	8.4%	0.143	5.3%	0.138	1.5%	0.138	1.4%	0.149	9.5%	0.143	5.4%	0.139	1.9%
C2 phenanthrenes/anthracenes	0.028	18.6%	0.023	4.1%	0.023	1.3%	0.024	2.7%	0.026	8.7%	0.022	5.6%	0.024	3.1%	0.023	4.5%	0.022	7.1%
C3 phenanthrenes/anthracenes	0.040	12.7%	0.037	3.0%	0.039	9.7%	0.037	2.0%	0.038	5.8%	0.039	9.2%	0.039	9.4%	0.038	4.7%	0.038	6.2%
C4 phenanthrenes/anthracenes	0.036	10.2%	0.033	3.0%	0.035	8.7%	0.036	9.9%	0.035	9.4%	0.033	0.7%	0.034	6.7%	0.035	8.8%	0.033	1.0%
fluoranthene	0.258	5.4%	0.254	4.1%	0.251	2.8%	0.250	2.2%	0.255	4.4%	0.253	3.4%	0.253	3.4%	0.237	2.9%	0.240	1.7%
pyrene	0.264	12.8%	0.246	4.9%	0.256	9.4%	0.244	4.1%	0.245	4.6%	0.258	9.9%	0.257	9.4%	0.242	3.1%	0.245	4.6%
C1 fluoranthenes/pyrenes	0.133	8.0%	0.150	4.0%	0.151	4.7%	0.145	0.6%	0.141	2.6%	0.138	4.4%	0.157	9.0%	0.143	0.8%	0.136	6.0%
benz[a]anthracene	0.332	11.1%	0.312	4.4%	0.317	6.3%	0.323	8.1%	0.317	6.2%	0.300	0.5%	0.319	6.9%	0.319	6.7%	0.316	5.6%
chrysene	0.390	9.7%	0.384	8.0%	0.382	7.6%	0.376	5.8%	0.364	2.6%	0.385	8.3%	0.382	7.6%	0.370	4.1%	0.375	5.6%
C1 chrysenes	0.183	9.7%	0.180	8.0%	0.180	7.6%	0.177	5.8%	0.171	2.6%	0.181	8.3%	0.180	7.6%	0.174	4.1%	0.176	5.6%
C2 chrysenes	0.097	9.7%	0.096	8.0%	0.096	7.6%	0.094	5.8%	0.091	2.6%	0.096	8.3%	0.096	7.6%	0.092	4.1%	0.094	5.6%
C3 chrysenes	0.078	9.7%	0.077	8.0%	0.076	7.6%	0.075	5.8%	0.073	2.6%	0.077	8.3%	0.076	7.6%	0.074	4.1%	0.075	5.6%
C4 chrysenes	0.058	9.7%	0.058	8.0%	0.057	7.6%	0.056	5.8%	0.055	2.6%	0.058	8.3%	0.057	7.6%	0.055	4.1%	0.056	5.6%
benzo[b+k]fluoranthene	1.056	1.7%	0.952	8.3%	1.051	1.3%	0.999	3.8%	1.072	3.3%	1.126	8.5%	1.020	1.8%	1.057	1.8%	1.105	6.4%
benzo[e]pyrene	1.029	1.4%	0.941	7.3%	1.059	4.4%	0.941	7.2%	1.114	9.8%	1.100	8.4%	1.112	9.6%	1.036	2.1%	1.086	7.1%
benzo[a] pyrene	0.724	0.0%	0.692	4.4%	0.661	8.7%	0.698	3.7%	0.691	4.6%	0.679	6.2%	0.771	6.5%	0.686	5.3%	0.665	8.1%
perylene	0.770	0.2%	0.725	5.6%	0.729	5.0%	0.740	3.6%	0.724	5.8%	0.752	2.1%	0.807	5.2%	0.722	5.9%	0.731	4.8%
indeno[1,2,3-cd]pyrene	1.147	11.7%	1.106	7.6%	1.073	4.4%	1.118	8.8%	1.018	1.0%	1.010	1.7%	1.081	5.2%	1.101	7.2%	1.024	0.4%
dibenz[ah]anthracene	1.472	7.8%	1.272	6.9%	1.369	0.3%	1.266	7.3%	1.388	1.6%	1.266	7.3%	1.231	9.9%	1.345	1.5%	1.331	2.5%
benzo[ghi]perylene	1.597	9.8%	1.538	5.7%	1.492	2.5%	1.476	1.4%	1.480	1.7%	1.513	4.0%	1.476	1.4%	1.440	1.0%	1.407	3.3%



Enclosure G

**Appendix A to the
Milwaukee Solvay Coke
and Gas Plant Site
Engineering Evaluation
and Cost Analysis Support
Sampling Plan, Addendum
to USEPA-Approved Multi-
Site Quality Assurance
Project Plan (NRT,
October 2017)**



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5

77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF: SR-6J

October 18, 2017

(via email)

We Energies

Robert Paulson, Principal Environmental Consultant
c/o WEC Business Services, LLC
333 West Everett St. A231
Milwaukee, WI 53203

RE: EPA Approval of Quality Assurance Project Plan Addendum, Appendix A to the EE/CA Support Sampling Plan, OU 2, Solvay Coke and Gas Company Site, Milwaukee, WI

Dear Mr. Paulson:

The U.S. Environmental Protection Agency (EPA) has completed a review of the Quality Assurance Project Plan (QAPP) Addendum submitted on September 25th, 2017, and revised on October 13th, 2017 as Appendix A to the Engineering Evaluation/Cost Analysis (EE/CA) Support Sampling Plan prepared in support of the EE/CA development at Operable Unit 2 (OU 2) of the Solvay Coke and Gas Company Site, located in Milwaukee, WI.

The purpose of this letter is to transmit EPA approval of the subject QAPP Addendum. Please provide us with a final copy of the QAPP Addendum, including a signature page, for our records.

If you have any questions I can be reached at by telephone at (312) 886-6943 or by email at patel.viral@epa.gov.

Sincerely,

A handwritten signature in blue ink, appearing to read "V. Patel", is positioned below the word "Sincerely,".

Ms. Viral Patel
Remedial Project Manager
Superfund Division

ecc:

Alida Roberman, EPA
Owen Thompson, EPA
Margaret Brunette, WDNR

Rick Mehl, Weston Solutions, Inc.
Jennifer Hagen, Natural Resource Technology
Jay Karls, Natural Resource Technology

ADDENDUM TO USEPA-APPROVED MULTI-SITE QUALITY ASSURANCE PROJECT PLAN

This Addendum provides site-specific elements as identified in the USEPA-Approved Multi-site Quality Assurance Project Plan (QAPP). Unless specifically noted otherwise, the Multi-site QAPP prepared for WEC Energy Group's Wisconsin Public Service Corporation's (WPSC) former MGP sites being addressed in an AOC for Remedial Investigations and Feasibility Studies (RI/FS), CERCLA Docket No. V-W-06-C-847, effective May 5, 2006, will be followed while implementing the Engineering Evaluation and Cost Analysis (EE/CA) Support Sampling Plan at the Milwaukee Solvay Coke and Gas Site (Site), located in Milwaukee, Wisconsin. The Multi-site QAPP is reviewed internally on an annual basis or as additional elements are required for the program. As a result of the 2017 review, an addendum to the Multi-site QAPP is anticipated to be submitted by early November 2017, to expand existing lab's analytical capabilities. Remaining sections of the Multi-site QAPP continue to be applicable. This appendix references the Multi-site QAPP sections which may require site-specific elements.

SECTION 1.1 – INTRODUCTION

The Multi-site QAPP addresses all the activities to be performed at the Site. Updates to reporting limits, method detection limits and laboratory standard operating procedures for the selected analytical laboratories, Pace Analytical Services, LLC and STAT Analysis Corporation, are included herein as discussed below (Section 1.4).

SECTION 1.2 – PROJECT TASK ORGANIZATION

The lines of authority specific to Milwaukee Solvay Coke and Gas Site are presented in Figure 1.

SECTION 1.3 – PROBLEM DEFINITION/BACKGROUND INFORMATION (A5)

The EE/CA Support Sampling Plan has been prepared to further determine the nature and extent of source material and address any remaining data gaps at the (Site) located in Milwaukee, Wisconsin. Specifically, this EE/CA Support Sampling Plan addresses data acquisition activities necessary to evaluate upland area removal alternatives in the EE/CA and to support a Non-Time Critical Removal Action (NTCRA) at the Site.

As discussed in the USEPA-approved Multi-site QAPP as typical field activities, the Milwaukee Solvay Coke and Gas Plant investigation will include test pits, surface and subsurface soil sampling (analytical and/or visual observations), and groundwater sampling. Previously collected data (as summarized in Sections 2.6, 2.7, and 2.8 of the EE/CA Support Sampling) have been used to evaluate the need for and location of additional sampling. The Problem Statement for the Site is refined to:

To determine the current nature and extent of non-aqueous phase liquid (NAPL) in soil and characterize groundwater and surface soil quality in preparation for a Non-Time Critical Removal Action.

SECTION 1.4 – PROJECT/TASK DESCRIPTION AND SCHEDULE (A6)

Conditions that require further assessment to support an EE/CA with the remedial action objectives (RAOs) as presented in the AOC (Docket # V-W-17-C-010, effective August 31, 2017) are presented in Section 3.2 of the EE/CA Support Sampling Plan. Activities include, soil and groundwater sampling.

The other activities presented in Section 1.4 of the Multi-site QAPP are not anticipated at this time.

Site-specific tasks to be performed and the sampling rationale are presented in Section 3 and Table 3 of the EE/CA Support Sampling Plan.

The project quantitation limits (PQLs) for the EE/CA Support Sampling Plan are provided as an attachment on Uniform Federal Policy (UFP) Worksheet #15. Pace Analytical Services is proposed for soil and water analyses. Alpha is proposed for select soil sample analysis. Both laboratories are included in the USEPA approved Multi-Site QAPP. Updates to the laboratory's practical quantitation limit (PQL) and reporting limits (RL) compared to applicable Site screening levels are also provided on UFP Worksheet #15. A site-specific sampling and analysis

summary is presented on Table 4 of the EE/CA Support Sampling Plan and Table 3 and 4 detail the media to be sampled and the constituents to be analyzed.

A site-specific schedule for implementing the EE/CA Support Sampling Plan activities is presented in Section 4 of the EE/CA Support Sampling Plan.

SECTION 1.5.1 – STEP 1 PROBLEM STATEMENT

Team members and roles are identified in Figure 1 and Section 1.2 of the Multi-Site QAPP. As discussed above, the Problem Statement for the Site is refined to:

To determine the current nature and extent of non-aqueous phase liquid (NAPL) in soil and characterize groundwater and surface soil quality in preparation for a Non-Time Critical Removal Action.

SECTION 1.5.2 – STEP 2 DECISION IDENTIFICATION

The study results will provide data to support a Non-Time Critical Removal Action. The objectives of the study are provided in Section 3.2 of the EE/CA Support Sampling Plan and summarized below:

- Refine the extent of NAPL and cyanide in surface and subsurface soil at the site
- Evaluate the depth to peat and clay layers
- Characterize pre-removal groundwater quality
- Characterize direct contact zone soils to support an industrial land use
- Sewer and water intake inspections to document current conditions

These data will be used to support a removal action approach, anticipated to include excavation or in-situ treatment, a surface barrier and institutional controls as discussed in Section 27 of the AOC and summarized in Section 3.1 of the EE/CA Support Sampling Plan.

SECTION 1.5.3 – STEP 3 DECISION INPUTS

As mentioned above, Tables 3 and 4 summarize the media to be sampled, rationale, and the constituents to be analyzed. The laboratory's PQL and RL compared to applicable Site screening levels are provided on UFP Worksheet #15. In some instances, the PQL may be above the screening level because commercially available techniques cannot achieve detection levels below the screening levels.

The proposed sampling and analysis plan summary is provided on Table 3 of the EE/CA Support Sampling Plan.

The analyte 1,2-Dibromo-3-chloropropane reporting limit exceeds the screening level for direct contact but was not identified as a COC in the Remedial Investigation/Feasibility Study Group (RI/FS) RI Report (Arcadis, 2016). The detection limit is below the screening level for direct contact and therefore, these data will be estimated for the EE/CA Support Sampling.

Soil to groundwater values are calculated based on Wisconsin groundwater standards, and are used as indicators that further evaluation of groundwater is warranted. Wisconsin Department of Natural Resources (WDNR) recognizes that calculated values are not achievable by laboratories, and detection limits and reporting limits presented in the attached Worksheet #15 are consistent with WDNR published RL and DL laboratory requirements for assessments under NR 700.

Consistent with WDNR policy, the historical site use of the property where potential barrier fill material will be generated will be reviewed in addition to soil analytical sampling. If the historical site use identifies the potential for compounds to be present, synthetic precipitation leaching procedure (SPLP) analysis will be performed and the QAPP will be amended at that time.

Analytes with reporting limits and detection limits exceeding groundwater screening levels exist but were not identified as COCs in the RI/FS Group RI Report (Arcadis, 2016). In some instances, the detection limit is below the screening level for groundwater and therefore, these data will be estimated for the EE/CA Support Sampling. In addition, the intent of the groundwater sampling event is to characterize pre-remediation groundwater quality. Groundwater remedial objectives will be established in a separate effort, outside of the EE/CA and Non-Time Critical Removal.

Section 3 and Appendix J of the EE/CA Support Sampling Plan describe the standard operating procedures (SOPs) to be used in the field. The laboratories' SOPs are attached.

SECTION 1.5.3.1 – SCREENING LEVEL ECOLOGICAL RISK ASSESSMENT (SLERA)

An SLERA was prepared as part of the USEPA-approved Remedial Investigation Report, prepared for Milwaukee Solvay Coke and Gas Site Remedial Investigation/Feasibility Study Group and an update is not anticipated to be necessary.

SECTION 1.5.3.2 – HUMAN HEALTH RISK ASSESSMENT (HHRA)

An HHRA was prepared as part of the USEPA-approved Remedial Investigation Report, prepared for Milwaukee Solvay Coke and Gas Site Remedial Investigation/Feasibility Study Group and an update is not anticipated to be necessary.

SECTION 1.5.4 – STEP 4 INVESTIGATION BOUNDARIES

Figure 2 of the EE/CA Sampling Support Work Plan depicts the site boundaries to be investigated. Figures 8a and 8b provide proposed sampling locations.

Sample volumes required for laboratory and toxicity testing are provided on Table 4 of the EE/CA Sampling Support Work Plan.

As mentioned above, the sampling rationale is presented in Section 3 and Table 3 of the EE/CA Sampling Support Work Plan.

SECTION 1.5.5 – STEP 5 DECISION RULES

The need for a removal action has been established per the AOC (V-W-17-C-010). Based on this study, if NAPL/cyanide is encountered outside of the footprints previously identified, the removal footprint will be expanded. If NAPL/cyanide is not encountered inside the footprints previously identified, the removal footprint may contract.

If other conditions are encountered that threaten human health or the environment, these conditions will be abated or mitigated to the extent possible in a Non-Time Critical Removal Action.

SECTION 1.5.6 – STEP 6 DECISION ERROR LIMITS

Sampling design and laboratory analyses errors will be addressed as described in Section 1.5.6 of the USEPA-approved Multi-site QAPP.

SECTION 1.5.7 – STEP 7 OPTIMIZING DESIGN

Optimizing the design in the upland areas is described in Section 1.5.7 of the USEPA-approved Multi-site QAPP. The river sediment and surface water are not included in this scope of work.

SECTION 1.5.8 – MEASUREMENT PERFORMANCE CRITERIA

The overall objectives and criteria for assuring quality for this effort are described in Section 1.5.8 of the USEPA-approved Multi-site QAPP. The laboratories selected to perform the analysis are discussed in Section 3 of the EE/CA Sampling Support Work Plan and include Pace Analytical Services for soil and water samples and Alpha

for select soil samples. Both laboratories are included in the USEPA-approved Multi-site QAPP. Modifications, if any, will be provided to USEPA for review and approval in accordance with the AOC/SOW.

SECTION 1.6 AND SECTION 1.7

Section 1.6 and Section 1.7 will follow the USEPA-approved Multi-site QAPP.

SECTION 2.1.1 – SCHEDULE

A site-specific schedule for implementing the EE/CA Sampling Support Work Plan is presented in Section 4 of the EE/CA Sampling Support Work Plan.

SECTION 2.1.2 – SAMPLING DESIGN RATIONALE

Table 3 provides sample location rationale and target depths. Table 4 summarizes the media to be sampled and the constituents to be analyzed.

SECTION 2.2 – SAMPLING METHODS REQUIREMENTS

Section 3 and Appendix J of the EE/CA Support Sampling Plan describe the standard operating procedures (SOPs) to be used in the field and are consistent with the USEPA-approved Multi-site QAPP.

SECTION 2.3 – SAMPLING HANDLING AND CUSTODY REQUIREMENTS

Section 3 and Appendix J of the EE/CA Support Sampling Plan describe the standard operating procedures (SOPs) to be used in the field and are consistent with the USEPA-approved Multi-site QAPP. Analytical laboratory SOPs are attached.

SECTION 2.3.1.3 – SAMPLE CONTAINER, VOLUME, PRESERVATION AND HOLDING TIME

Table 4 summarizes the sample containers, volume, preservation and holding times for the media to be sampled and the constituents to be analyzed.

SECTION 2.4 – ANALYTICAL METHODS REQUIREMENTS (B4)

Table 4 summarizes the parameters to be analyzed.

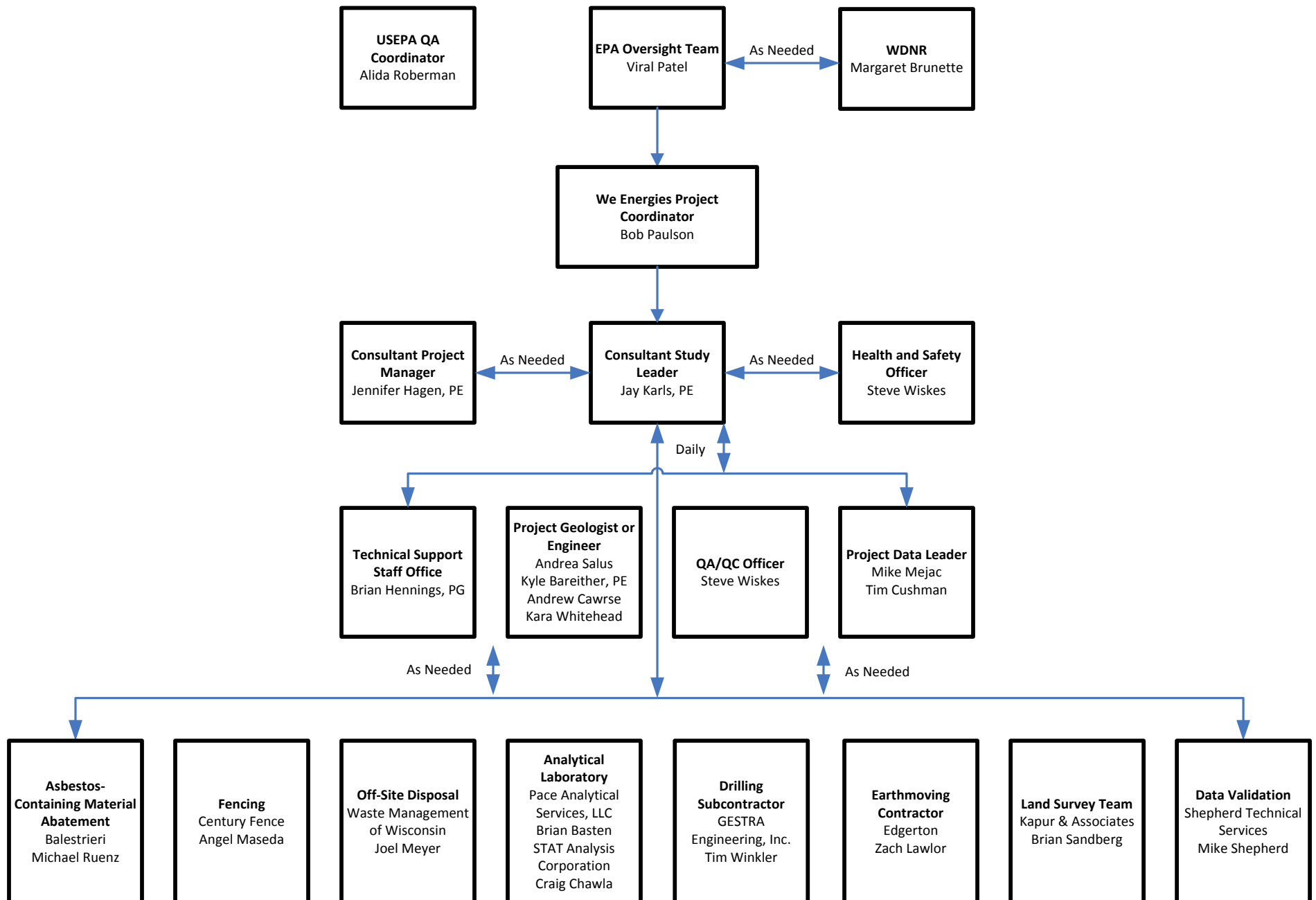
REMAINING QAPP SECTIONS

Remaining QAPP sections will follow the intent of the USEPA-approved Multi-site QAPP.

ATTACHMENTS:

Figure 1	Lines of Communication
Attachment 1	QAPP Worksheet #15
Attachment 2	Analytical Laboratory Standard Operating Procedures
Attachment 2-1	Pace Analytical Services, LLC
Attachment 2-2	STAT Analysis Corporation

FIGURE 1 - MILWAUKEE SOLVAY COKE & GAS SITE TECHNICAL TEAM STRATEGY





Attachment 1
QAPP Worksheet #15

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil

Analytical Method: 8260B

Concentration level (if applicable):

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Volatile Organic Compounds by Method SW846-8260B					
Acetone	67-64-1	100000	3.677		
Acetonitrile	75-05-8	4920	#N/A		
Acrolein (Propenal)	107-02-8	0.873	#N/A		
Acrylonitrile	107-13-1	1.5	#N/A		
Allyl alcohol	107-18-6	21.5	#N/A		
Allyl chloride	107-05-1	4.54	#N/A		
Benzene	71-43-2	7.07	0.005	0.025	0.060
Benzyl chloride	100-44-7	6.23	#N/A		
Bis(2-chloroethyl)sulfide	505-60-2	1050	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Bromoacetone	598-31-2	#N/A	#N/A		
Bromochloromethane	74-97-5	906	#N/A	0.025	0.060
Bromodichloromethane	75-27-4	1.83	0.0003	0.025	0.060
4-Bromofluorobenzene (surr)	460-00-4	323	#N/A		
Bromoform	75-25-2	113	0.002	0.025	0.060
Bromomethane	74-83-9	43	0.005	0.069093	0.250
n-Butanol	71-36-3	7640	#N/A		
2-Butanone (MEK)	78-93-3	28400	1.6661		
t-Butyl alcohol	75-65-0	#N/A	0.0049		
Carbon disulfide	75-15-0	738	0.5919		
Carbon tetrachloride	56-23-5	4.03	0.0039	0.025	0.060
Chloral hydrate	302-17-0	100000	#N/A		
Chlorobenzene	108-90-7	761	0.1358	0.025	0.060
Chlorobenzene-d ₅ (IS)	3114-55-4	#N/A	#N/A		
Chlorodibromomethane	124-48-1	38.9	0.032	0.025	0.060
Chloroethane	75-00-3	2120	0.227	0.067017716	0.250
2-Chloroethanol	107-07-3	23400	#N/A		
2-Chloroethyl vinyl ether	110-75-8	117	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Chloroform	67-66-3	1.98	0.003	0.046442419	0.250
Chloromethane	74-87-3	669	0.016	0.025	0.060
Chloroprene	126-99-8	0.0636	#N/A		
3-Chloropropionitrile	542-76-7	#N/A	#N/A		
Crotonaldehyde	4170-30-3	20100	#N/A		
1,2-Dibromo-3-chloropropane	96-12-8	0.0923	0.00017	0.091241581	0.250
1,2-Dibromoethane	106-93-4	0.221	0.00003	0.025	0.060
Dibromomethane	74-95-3	143	#N/A	0.025	0.060
1,2-Dichlorobenzene	95-50-1	376	1.168	0.025	0.060
1,3-Dichlorobenzene	541-73-1	297	1.153	0.025	0.060
1,4-Dichlorobenzene	106-46-7	16.4	0.144	0.025	0.060
1,4-Dichlorobenzene-d ₄ (IS)	3855-82-1	#N/A	#N/A		
cis-1,4-Dichloro-2-butene	1476-11-5	0.0469	#N/A		
trans-1,4-Dichloro-2-butene	110-57-6	0.0469	#N/A		
Dichlorodifluoromethane	75-71-8	530	3.0863	0.025	0.060
1,1-Dichloroethane	75-34-3	22.2	0.4834	0.025	0.060
1,2-Dichloroethane	107-06-2	2.87	0.0028	0.025	0.060
1,2-Dichloroethane-d ₄ (surr)	17060-07-0	#N/A	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
1,1-Dichloroethene	75-35-4	1190	0.0050	0.025	0.060
trans-1,2-Dichloroethene	156-60-5	1850	0.0626	0.025	0.060
1,2-Dichloropropane	78-87-5	1.78	0.0033	0.025	0.060
1,3-Dichloro-2-propanol	96-23-1	#N/A	#N/A		
cis-1,3-Dichloropropene	10061-01-5	1210	#N/A	0.025	0.060
trans-1,3-Dichloropropene	10061-02-6	1510	#N/A	0.025	0.060
1,2,3,4-Diepoxybutane	1464-53-5	100000	#N/A		
Diethyl ether	60-29-7	10100	0.4478		
1,4-Difluorobenzene (IS)	540-36-3	#N/A	#N/A		
1,4-Dioxane	123-91-1	26.5	0.0012		
Epichlorohydrin	106-89-8	117	#N/A		
Ethanol	64-17-5	100000	#N/A		
Ethyl acetate	141-78-6	3800	#N/A		
Ethylbenzene	100-41-4	35.4	1.57	0.025	0.060
Ethylene oxide	75-21-8	0.0358	#N/A		
Ethyl methacrylate	97-63-2	1100	#N/A		
Fluorobenzene (IS)	462-06-6	2390	#N/A		
Hexachlorobutadiene	87-68-3	7.19	#N/A	0.025	0.060

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Hexachloroethane	67-72-1	11.1	#N/A		
2-Hexanone	591-78-6	1760	#N/A		
2-Hydroxypropionitrile	78-97-7	#N/A	#N/A		
Iodomethane	74-88-4	3040	#N/A		
Isobutyl alcohol	78-83-1	10000	#N/A		
Isopropylbenzene	98-82-8	268	#N/A	0.025	0.060
Malononitrile	109-77-3	82.1	#N/A		
Methacrylonitrile	126-98-7	107	#N/A		
Methanol	67-56-1	100000	2.0245		
Methylene chloride	75-09-2	1150	0.0026	0.025	0.060
Methyl methacrylate	80-62-6	2360	#N/A		
4-Methyl-2-pentanone (MIBK)	108-10-1	3360	0.2252		
Naphthalene	91-20-3	24.1	0.6582	0.0400453	0.250
Nitrobenzene	98-95-3	32.4	#N/A		
2-Nitropropane	79-46-9	0.0861	#N/A		
N-Nitroso-di-n-butylamine	924-16-3	0.494	#N/A		
Paraldehyde	123-63-7	#N/A	#N/A		
Pentachloroethane	76-01-7	36.3	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
2-Pentanone	107-87-9	#N/A	#N/A		
2-Picoline	109-06-8	100000	#N/A		
1-Propanol	71-23-8	100000	#N/A		
2-Propanol	67-63-0	34500	#N/A		
Propargyl alcohol	107-19-7	2340	#N/A		
3-Propiolactone	57-57-8	#N/A	#N/A		
Propionitrile (ethyl cyanide)	107-12-0	15600	#N/A		
n-Propylamine	107-10-8	#N/A	#N/A		
Pyridine	110-86-1	1170	0.0069		
Styrene	100-42-5	867	0.22	0.025	0.060
1,1,1,2-Tetrachloroethane	630-20-6	12.3	0.0534	0.025	0.060
1,1,2,2-Tetrachloroethane	79-34-5	3.6	0.0002	0.025	0.060
Tetrachloroethene	127-18-4	145	0.0045	0.025	0.060
Toluene	108-88-3	818	1.1072	0.025	0.060
Toluene-d8 (surr)	2037-26-5	#N/A	#N/A		
o-Toluidine	95-53-4	144	#N/A		
1,2,4-Trichlorobenzene	120-82-1	113	0.408	0.047550292	0.250
1,1,1-Trichloroethane	71-55-6	640	0.1402	0.025	0.060

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
1,1,2-Trichloroethane	79-00-5	7.01	0.0032	0.025	0.060
Trichloroethene	79-01-6	8.41	0.0036	0.025	0.060
Trichlorofluoromethane	75-69-4	1230	4.4775	0.025	0.060
1,2,3-Trichloropropane	96-18-4	0.109	0.0519	0.025	0.060
Vinyl acetate	108-05-4	2750	#N/A		
Vinyl chloride	75-01-4	2.08	0.0001	0.025	0.060
o-Xylene	95-47-6	434	#N/A	0.025	0.060
m-Xylene	108-38-3	388	#N/A		
p-Xylene	106-42-3	390	#N/A		
1,3,5-Trimethylbenzene	108-68-8	182	#N/A	0.025	0.060
1,2,4-Trimethylbenzene	95-63-6	219	#N/A	0.025	0.060

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Soil

Analytical Method: 8270C PAHs by 8270SIM

Concentration level (if applicable):

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Semi-volatile Organic Compounds by Method SW846-8270C (PAHs by 8270 SIM)					
Acenaphthene	83-32-9	45200	#N/A	0.00388	0.0129
Acenaphthylene	208-96-8	#NA	#N/A	0.0330	0.0110
Acetophenone	98-86-2	2520	#N/A		
2-Acetylaminofluorene	53-96-3	0.605	#N/A		
1-Acetyl-2-thiourea	591-08-2	#N/A	#N/A		
2-Aminoanthraquinone	117-79-3	#N/A	#N/A		
Aminoazobenzene	60-09-3	#N/A	#N/A		
4-Aminobiphenyl	92-67-1	0.109	#N/A		
3-Amino-9-ethylcarbazole	132-32-1	#N/A	#N/A		
Anilazine	101-05-3	#N/A	#N/A		
Aniline	62-53-3	403	#N/A		
o-Anisidine	90-04-0	#N/A	#N/A		
Anthracene	120-12-7	100000	196.95	0.00571	0.0190
Aramite	140-57-8	91.9	#N/A		
Azinphos-methyl	86-50-0	2460	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Barban	101-27-9	#N/A	#N/A		
Benzidine	92-87-5	0.00999	#N/A		
Benzoic Acid	65-85-0	100000	#N/A		
Benz(a)anthracene	56-55-3	20.8	#N/A	0.00317	0.0106
Benzo(b)fluoranthene	205-99-2	21.1	0.4793	0.00282	0.00941
Benzo(k)fluoranthene	207-08-9	211	#N/A	0.00251	0.00836
Benzo(g,h,i)perylene	191-24-2	#N/A	#N/A	0.00203	0.00677
Benzo(a)pyrene	50-32-8	2.11	0.47	0.00251	0.00837
Benzo(e)pyrene	192-97-2	#N/A	#N/A		
p-Benzoquinone	106-51-4	#N/A	#N/A		
Benzyl alcohol	100-51-6	82100	#N/A		
Bis(2-chloroethoxy)methane	111-91-1	2460	#N/A	0.0449525	0.14984
Bis(2-chloroethyl) ether	111-44-4	1.29	#N/A	0.0521093	0.1737
Bis(2-chloroisopropyl) ether	108-60-1	1020	#N/A		
Bis(2-ethylhexyl) phthalate	117-81-7	164	2.88	0.0277559	0.09252
4-Bromophenyl phenyl ether	101-55-3	26.9	#N/A	0.0349569	0.11652
Bromoxynil	1689-84-5	22.3	#N/A		
Butyl benzyl phthalate	85-68-7	1210	#N/A	0.0267668	0.08922

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Captafol	2425-06-1	15.3	#N/A		
Captan	133-06-2	999	#N/A		
Carbaryl	63-25-2	82100	0.0726		
Carbofuran	1563-66-2	4100	0.0312		
Carbophenothion	786-19-6	#N/A	#N/A		
Chlordane (NOS)	57-74-9	#N/A	#N/A		
Chlorfenvinphos	470-90-6	574	#N/A		
4-Chloroaniline	106-47-8	11.5	#N/A	0.0274314	0.09143
Chlorobenzilate	510-15-6	20.9	#N/A		
5-Chloro-2-methylaniline	95-79-4	#N/A	#N/A		
4-Chloro-3-methylphenol	59-50-7	82100	#N/A	0.0519369	0.17312
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	#N/A	#N/A		
1-Chloronaphthalene	90-13-1	266	#N/A		
2-Chloronaphthalene	91-58-7	60300	#N/A	0.0214304	0.071434
2-Chlorophenol	95-57-8	5840	#N/A	0.0416619	0.13887
4-Chloro-1,2-phenylenediamine	95-83-0	#N/A	#N/A		
4-Chloro-1,3-phenylenediamine	5131-60-2	#N/A	#N/A		
<u>4-Chlorophenyl phenyl ether</u>	7005-72-3	#N/A	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
<u>Chrysene</u>	218-01-9	2110	0.1446	0.00337	0.0112
<u>Coumaphos</u>	56-72-4	#N/A	#N/A		
<u>p-Cresidine</u>	120-71-8	#N/A	#N/A		
<u>Crotoxyphos</u>	7700-17-6	#N/A	#N/A		
<u>2-Cyclohexyl-4,6-dinitro-phenol</u>	131-89-5	1640	#N/A		
<u>Demeton-O</u>	298-03-3	#N/A	#N/A		
<u>Demeton-S</u>	126-75-0	#N/A	#N/A		
<u>Diallate (cis or trans)</u>	2303-16-4	37.7	#N/A		
<u>2,4-Diaminotoluene</u>	95-80-7	#N/A	#N/A		
<u>Dibenz(a,j)acridine</u>	224-42-0	#N/A	#N/A		
<u>Dibenz(a,h)anthracene</u>	53-70-3	2.11	#N/A	0.00224	0.00745
<u>Dibenzofuran</u>	132-64-9	1040	#N/A	0.0202055	0.06735
<u>Dibenzo(a,e)pyrene</u>	192-65-4	0.176	#N/A		
<u>1,2-Dibromo-3-chloropropane</u>	96-12-8	0.0923	0.0002		
<u>Di-n-butyl phthalate</u>	84-74-2	82100	5.0333	0.0249484	0.08316
<u>Dichlone</u>	117-80-6	#N/A	#N/A		
<u>1,2-Dichlorobenzene</u>	95-50-1	376	1.168	0.052485	0.17495
<u>1,3-Dichlorobenzene</u>	541-73-1	297	1.1528	0.0231143	0.077047

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
<u>1,4-Dichlorobenzene</u>	106-46-7	16.4	0.144	0.023254	0.077513
<u>3,3'-Dichlorobenzidine</u>	91-94-1	5.11	#N/A	0.0452872	0.15096
<u>2,4-Dichlorophenol</u>	120-83-2	2460	#N/A	0.0446065	0.14868
<u>2,6-Dichlorophenol</u>	87-65-0	#N/A	#N/A		
<u>Dichlorovos</u>	62-73-7	7.92	#N/A		
<u>Dicrotophos</u>	141-66-2	57.4	#N/A		
<u>Diethyl phthalate</u>	84-66-2	100000	#N/A	0.0276781	0.09226
<u>Diethylstilbestrol</u>	56-53-1	0.00657	#N/A		
<u>Diethyl sulfate</u>	64-67-5	#N/A	#N/A		
<u>Dimethoate</u>	60-51-5	1810	0.00090		
<u>3,3'-Dimethoxybenzidine</u>	119-90-4	1.44	#N/A		
<u>Dimethylaminoazobenzene</u>	60-11-7	0.5	#N/A		
<u>7,12-Dimethylbenz(a)-anthracene</u>	57-97-6	0.0084	#N/A		
<u>3,3'-Dimethylbenzidine</u>	119-93-7	0.209	#N/A		
<u>α,α-Dimethylphenethylamine</u>	122-09-8	#N/A	#N/A		
<u>2,4-Dimethylphenol</u>	105-67-9	16400	#N/A	0.0330074	0.11002
<u>Dimethyl phthalate</u>	131-11-3	7390	#N/A	0.0217128	0.07238
<u>1,2-Dinitrobenzene</u>	528-29-0	82.1	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
1,3-Dinitrobenzene	99-65-0	82.1	#N/A		
1,4-Dinitrobenzene	100-25-4	82.1	#N/A		
4,6-Dinitro-2-methylphenol	534-52-1	65.7	#N/A	0.0514485	0.17149
2,4-Dinitrophenol	51-28-5	1640	#N/A	0.0508491	0.16949
2,4-Dinitrotoluene	121-14-2	7.37	0.00014	0.0238714	0.07957
2,6-Dinitrotoluene	606-20-2	1.54	0.00014	0.0316886	0.105628
Dinocap	39300-45-3	#N/A	#N/A		
Dinoseb	88-85-7	821	0.123		
Diphenylamine	122-39-4	82100	#N/A		
5,5-Diphenylhydantoin	57-41-0	#N/A	#N/A		
1,2-Diphenylhydrazine	122-66-7	2.87	#N/A		
Di-n-octyl phthalate	117-84-0	8210	#N/A	0.0375306	0.1251
Disulfoton	298-04-4	32.8	#N/A		
EPN	2104-64-5	8.21	#N/A		
Ethion	563-12-2	410	#N/A		
Ethyl carbamate	51-79-6	2.3	#N/A		
Ethyl methanesulfonate	62-50-0	#N/A	#N/A		
Famphur	52-85-7	#N/A	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Fensulfothion	115-90-2	#N/A	#N/A		
Fenthion	55-38-9	#N/A	#N/A		
Fluchloralin	33245-39-5	#N/A	#N/A		
Fluoranthene	206-44-0	30100	88.88	0.00521	0.0174
Fluorene	86-73-7	30100	14.83	0.00414	0.0138
2-Fluorobiphenyl (surr)	321-60-8	#N/A	#N/A		
2-Fluorophenol (surr)	367-12-4	27300	#N/A		
Hexachlorobenzene	118-74-1	1.15	0.0252	0.0280731	0.09358
Hexachlorobutadiene	87-68-3	7.19	#N/A		
Hexachlorocyclopentadiene	77-47-4	10.8	#N/A	0.0395002	0.13167
Hexachloroethane	67-72-1	11.1	#N/A	0.0267118	0.08904
Hexachlorophene	70-30-4	246	#N/A		
Hexachloropropene	1888-71-7	43.8	#N/A		
Hexamethylphosphoramide	680-31-9	328	#N/A		
Hydroquinone	123-31-9	38.3	#N/A		
Indeno(1,2,3-cd)pyrene	193-39-5	21.1	#N/A	0.00220	0.00733
Isodrin	465-73-6	#N/A	#N/A		
Isophorone	78-59-1	2420	#N/A	0.0256594	0.08553

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Isosafrole	120-58-1	234	#N/A		
Kepone	143-50-0	0.23	#N/A		
Leptophos	21609-90-5	#N/A	#N/A		
Malathion	121-75-5	16400	#N/A		
Maleic anhydride	108-31-6	80700	#N/A		
Mestranol	72-33-3	#N/A	#N/A		
Methapyrilene	91-80-5	#N/A	#N/A		
3-Methylcholanthrene	56-49-5	0.104	#N/A		
4,4'-Methylenebis(2-chloroaniline)	101-14-4	23	#N/A		
4,4'-Methylenebis(N,N-dimethyl-aniline)	101-61-1	50	#N/A		
Methyl methanesulfonate	66-27-3	23.2	#N/A		
2-Methylnaphthalene	91-57-6	3010	#N/A	0.00500	0.0167
Methyl parathion	298-00-0	205	#N/A		
2-Methylphenol	95-48-7	41000	#N/A	0.0303264	0.10109
3-Methylphenol	108-39-4	41000	#N/A		
4-Methylphenol	106-44-5	82100	#N/A		
Mevinphos	7786-34-7	#N/A	#N/A		
Mexacarbate	315-18-4	#N/A	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Mirex	2385-85-5	0.171	#N/A		
Monocrotophos	6923-22-4	#N/A	#N/A		
Naled	300-76-5	2340	#N/A		
Naphthalene	91-20-3	24.1	0.658	0.00842	0.0281
1,4-Naphthoquinone	130-15-4	#N/A	#N/A		
1-Naphthylamine	134-32-7	#N/A	#N/A		
2-Naphthylamine	91-59-8	1.28	#N/A		
Nicotine	54-11-5	#N/A	#N/A		
5-Nitroacenaphthene	602-87-9	#N/A	#N/A		
2-Nitroaniline	88-74-4	8010	#N/A	0.047567	0.15855
3-Nitroaniline	99-09-2	#N/A	#N/A	0.0283864	0.09462
4-Nitroaniline	100-01-6	115	#N/A	0.0692796	0.23093
5-Nitro-o-anisidine	99-59-2	46.9	#N/A		
Nitrobenzene	98-95-3	32.4	#N/A		
4-Nitrobiphenyl	92-93-3	#N/A	#N/A		
Nitrofen	1836-75-5	#N/A	#N/A		
2-Nitrophenol	88-75-5	#N/A	#N/A	0.0526808	0.1756
4-Nitrophenol	100-02-7	#N/A	#N/A	0.0420302	0.1401

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
5-Nitro-o-toluidine	99-55-8	255	#N/A		
Nitroquinoline-1-oxide	56-57-5	#N/A	#N/A		
N-Nitrosodi-n-butylamine	924-16-3	0.494	#N/A		
N-Nitrosodiethylamine	55-18-5	0.0153	#N/A		
N-Nitrosodimethylamine	62-75-9	0.0397	#N/A		
N-Nitrosomethylethylamine	10595-95-6	0.103	#N/A		
N-Nitrosodiphenylamine	86-30-6	469	0.076	0.2264811	0.75494
N-Nitrosodi-n-propylamine	621-64-7	0.328	#N/A	0.026474	0.08824
N-Nitrosomorpholine	59-89-2	0.343	#N/A		
N-Nitrosopiperidine	100-75-4	0.244	#N/A		
N-Nitrosopyrrolidine	930-55-2	1.09	#N/A		
Octamethyl pyrophosphoramidate	152-16-9	1640	#N/A		
4,4'-Oxydianiline	101-80-4	#N/A	#N/A		
Parathion	56-38-2	4920	#N/A		
Pentachlorobenzene	608-93-5	934	#N/A		
Pentachloronitrobenzene	82-68-8	12.6	#N/A		
Pentachlorophenol	87-86-5	3.97	0.0028	0.0367601	0.12253
Perylene	198-55-0	#N/A	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Phenacetin	62-44-2	1040	#N/A		
Phenanthrene	85-01-8	#N/A	#N/A	0.0116	0.0388
Phenobarbital	50-06-6	#N/A	#N/A		
Phenol	108-95-2	100000	2.295	0.039612	0.13204
1,4-Phenylenediamine	106-50-3	821	#N/A		
Phorate	298-02-2	164	#N/A		
Phosalone	2310-17-0	#N/A	#N/A		
Phosmet	732-11-6	16400	#N/A		
Phosphamidon	13171-21-6	#N/A	#N/A		
Phthalic anhydride	85-44-9	100000	#N/A		
2-Picoline (2-Methylpyridine)	109-06-8	100000	#N/A		
Piperonyl sulfoxide	120-62-7	#N/A	#N/A		
Pronamide	23950-58-5	61500	#N/A		
Propylthiouracil	51-52-5	#N/A	#N/A		
Pyrene	129-00-0	22600	54.545	0.00451	0.0150
Resorcinol	108-46-3	#N/A	#N/A		
Safrole	94-59-7	10.4	#N/A		
Strychnine	57-24-9	246	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Sulfallate	95-06-7	#N/A	#N/A		
Terbufos	13071-79-9	29.2	#N/A		
1,2,4,5-Tetrachlorobenzene	95-94-3	350	#N/A		
2,3,4,6-Tetrachlorophenol	58-90-2	24600	#N/A		
Tetrachlorvinphos	961-11-5	95.7	#N/A		
Tetraethyl dithiopyrophosphate	3689-24-5	410	#N/A		
Tetraethyl pyrophosphate	107-49-3	#N/A	#N/A		
Thionazine	297-97-2	#N/A	#N/A		
Thiophenol (Benzenethiol)	108-98-5	1170	#N/A		
Toluene diisocyanate	584-84-9	38.5	#N/A		
o-Toluidine	95-53-4	144	#N/A		
1,2,4-Trichlorobenzene	120-82-1	113	0.408	0.0188696	0.062898
2,4,5-Trichlorophenol	95-95-4	82100	#N/A	0.0294842	0.09828
2,4,6-Trichlorophenol	88-06-2	209	#N/A	0.0254504	0.08483
Trifluralin	1582-09-8	425	0.4939		
2,4,5-Trimethylaniline	137-17-7	#N/A	#N/A		
Trimethyl phosphate	512-56-1	115	#N/A		
1,3,5-Trinitrobenzene	99-35-4	32400	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Tris(2,3-dibromopropyl) phosphate	126-72-7	1.34	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.
Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: [Soil](#)

Analytical Method: [8082](#)

Concentration level (if applicable):

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Polychlorinated Biphenyls by Method SW846-8082					
Aroclor 1016	12674-11-2	28	#N/A	0.025	0.050
Aroclor 1221	11104-28-2	0.883	#N/A	0.025	0.050
Aroclor 1232	11141-16-5	0.792	#N/A	0.025	0.050
Aroclor 1242	53469-21-9	0.972	#N/A	0.025	0.050
Aroclor 1248	12672-29-6	0.975	#N/A	0.025	0.050
Aroclor 1254	11097-69-1	0.988	#N/A	0.025	0.050
Aroclor 1260	11096-82-5	1	#N/A	0.025	0.050
Aroclor 1262	37324-23-5	#N/A	#N/A	0.025	0.050
Aroclor 1268	11100-14-4	#N/A	#N/A	0.025	0.050

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Soil

Analytical Method: Parent and Alkylated PAHs by GC/MS-SIM by Alpha Analytical, Westborough, MA

Concentration level (if applicable):

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Method: Parent and Alkylated PAHs by GC/MS-SIM					
Acenaphthene	83-32-9	45200	#N/A	0.000177	0.001
Acenaphthylene	208-96-8	#N/A	#N/A	0.000192	0.001
Anthracene	120-12-7	100000	196.95	0.000207	0.001
Benzo(a)anthracene	56-55-3	20.8	#N/A	0.000205	0.001
Benzo(a)pyrene	50-32-8	2.11	0.47	0.000287	0.001
Benzo(b)fluoranthene	205-99-2	21.1	0.4793	0.000261	0.001
Benzo(e)pyrene	192-97-2	#N/A	#N/A	0.000207	0.001
Benzo(g,h,i)perylene	191-24-2	#N/A	#N/A	0.000267	0.001
Benzo(k)fluoranthene	207-08-9	211	#N/A	0.000199	0.001
C1-benzo(a)anthracene/chrysenes (alkyl chrysenes)	---	#N/A	#N/A	0.000203	0.001
C1-fluorenes	---	#N/A	#N/A	0.000268	0.001
C1-naphthalenes	---	#N/A	#N/A	0.000289	0.001
C1-phenanthrene/anthracenes	---	#N/A	#N/A	0.000333	0.001

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
C1-pyrene/fluoranthenes	---	#N/A	#N/A	0.000264	0.001
C2-benzo(a)anthracene/chrysenes (alkyl chrysenes)	---	#N/A	#N/A	0.000203	0.001
C2-fluorenes	---	#N/A	#N/A	0.000268	0.001
C2-napthalenes	---	#N/A	#N/A	0.000289	0.001
C2-phenanthrene/anthracenes	---	#N/A	#N/A	0.000333	0.001
C3-benzo(a)anthracene/chrysenes (alkyl chrysenes)	---	#N/A	#N/A	0.000203	0.001
C3-fluorenes	---	#N/A	#N/A	0.000268	0.001
C3-napthalenes	---	#N/A	#N/A	0.000289	0.001
C3-phenanthrene/anthracenes	---	#N/A	#N/A	0.000333	0.001
C4-benzo(a)anthracene/chrysenes (alkyl chrysenes)	---	#N/A	#N/A	0.000203	0.001
C4-napthalenes	---	#N/A	#N/A	0.000289	0.001
C4-phenanthrene/anthracenes	---	#N/A	#N/A	0.000333	0.001
Carbazole	86-74-8	#N/A	#N/A	0.000329	0.001
Chrysene	218-01-9	2110	0.1446	0.000203	0.001
Dibenzo(a,h)anthracene (ah+ac)	53-70-3	2.11	#N/A	0.000271	0.001
Dibenzofuran	132-64-9	1040	#N/A	0.000316	0.001

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Fluoranthene	206-44-0	30100	88.88	0.000319	0.001
Fluorene	86-73-7	30100	14.83	0.000264	0.001
Indeno(1,2,3-cd)pyrene	193-39-5	21.1	#N/A	0.000273	0.001
1-Methylnaphthalene	90-12-0	72.7	#N/A	0.000317	0.001
2-Methylnaphthalene	91-57-6	3010	#N/A	0.000259	0.001
Naphthalene	91-20-3	24.1	0.658	0.000289	0.001
Perylene	198-55-0	#N/A	#N/A	0.000194	0.001
Phenanthrene	85-01-8	#N/A	#N/A	0.000333	0.001
Pyrene	129-00-0	22600	54.545	0.000264	0.001

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Soil

Analytical Method: As noted

Concentration level (if applicable):

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Inorganic Compounds by Method SW846-6020A					
Aluminum	7429-90-5	100000	600	78.789	262.613
Antimony	7440-36-0	467	0.542	0.165	0.667
Arsenic	7440-38-2	3	0.584	0.264	0.879
Barium	7440-39-3	100000	164.8	0.229	0.762
Beryllium	7440-41-7	2300	6.32	0.080	0.667
Cadmium	7440-43-9	985	0.752	0.097	0.667
Calcium	7440-70-2	#N/A	#N/A	185.528	618.424
Chromium, Total	7440-47-3	#N/A	360000	1.338	4.459
Chromium (III)	16065-83-1	100000	#N/A		
Chromium (VI)	18540-29-9	6.36	#N/A		
Cobalt	7440-48-4	347	3.6073	0.089	0.667
Copper	7440-50-8	46700	91.6	0.393	1.309
Cyanide (SW-846 9012A)	57-12-5	195	4.04	0.12000	0.40
Iron	7439-89-6	100000	#N/A	39.297	166.675
Lead	7439-92-1	800	27	0.181	0.667

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Magnesium	7439-95-4	#N/A	#N/A	20.696	166.675
Manganese	7439-96-5	25900	39.124	0.814	2.713
Mercury (SW-846 7471B)	7439-97-6	3.13	0.208	0.0110	0.0370
Nickel	7440-02-0	22500	13.061	0.263	0.877
Potassium	7440-09-7	#N/A	#N/A	310.310	1034.385
Selenium	7882-49-2	5840	0.52	0.182	0.667
Silver	7440-22-4	5840	0.849	0.095	0.333
Sodium	7440-23-5	#N/A	#N/A	9.956	166.675
Thallium	7440-28-0	11.7	0.284	0.114	0.667
Vanadium	7440-62-2	5840	60	0.428	1.428
Zinc	7440-66-6	100000	#N/A	3.650	12.167

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Soil

Analytical Method: 8270C

Concentration level (if applicable):

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Pesticides by Method SW846-8081					
Aldrin	309-00-2	0.187	#N/A	0.0005459	0.00182
α-BHC	319-84-6	0.365	#N/A	0.0004657	0.00155
β-BHC	319-85-7	1.28	#N/A	0.00054	0.0018
δ-BHC	319-86-8	#N/A	#N/A	0.000508	0.00169
γ-BHC (Lindane)	58-89-9	2.54	0.0023	0.001054	0.00351
α-Chlordane	5103-71-9	#N/A	#N/A	0.0005548	0.00185
γ-Chlordane	5566-34-7	#N/A	#N/A	0.0006387	0.00213
4,4'-DDD	72-54-8	9.57	#N/A	0.00102	0.0034
4,4'-DDE	72-55-9	9.38	#N/A	0.001085	0.00362
4,4'-DDT	50-29-3	8.53	#N/A	0.0016	0.00533
Dieldrin	60-57-1	0.144	#N/A	0.001047	0.00349
Endosulfan I	959-98-8	#N/A	#N/A	0.0005252	0.00175
Endosulfan II	33213-65-9	#N/A	#N/A	0.001328	0.00443
Endosulfan sulfate	1031-07-8	#N/A	#N/A	0.001284	0.00428
Endrin	72-20-8	246	0.1616	0.001177	0.00392

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI Industrial DC RCL (mg/kg)	WI Soil to GW RCL (mg/Kg)	Laboratory Detection Limit (mg/Kg)	Laboratory Reporting Limit (mg/Kg)
Endrin aldehyde	7421-93-4	#N/A	#N/A	0.001173	0.00391
Endrin ketone	53494-70-5	#N/A	#N/A	0.001745	0.00582
Heptachlor	76-44-8	0.654	0.0662	0.0005843	0.00195
Heptachlor epoxide	1024-57-3	0.338	0.0082	0.0004931	0.00164
Methoxychlor	72-43-5	4100	4.32	0.007521	0.0251
Toxaphene	8001-35-2	2.09	0.928	0.0141078	0.0470212974

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Soil

Analytical Method: As noted

Concentration level (if applicable):

Analyte	CAS No.	Project Action Limit	Laboratory Detection Limit	Laboratory Reporting Limit
Waste Characterization – Hazardous Characteristics				
Total Solids (A2540 B-97)	NA	NA (%)		
Density – Bulk (D5057-90)	NA	NA (lb/ft ³)		
Density – Bulk, Dry (D5057-90)	NA	NA (lb/ft ³)		
Density – Bulk, Wet (D5057-90)	NA	NA (lb/ft ³)		
Moisture (E160.3M)	NA	NA (%)		
Flashpoint/Ignitability (SW1010A)	NA	<140 °F		
Cyanide, Reactive (SW 7.3.3.2)	57-12-5	NA (mg/Kg)	0.4 mg/kg	1 mg/kg
Sulfide, Reactive (SW 7.3.4.2)	18496-25-8	NA (mg/Kg)	10 mg/kg	10 mg/kg
pH, corrosivity (SW9045D)	NA	2 < pH < 12.5	0.01 Std. Units	0.1 Std. Units
Chloride (SW9056A)	NA	NA (mg/Kg)		
Phenolics, Total (SW9066)	NA	NA (mg/Kg)		
Free Liquids (SW9095B)	NA	Pass		
Analyte	CAS No.	Project Action Limit (mg/L)	Laboratory Detection Limit (mg/L)	Laboratory Reporting Limit (mg/L)
Waste Characterization – Toxicity Characteristics by TCLP Method 1311				
TCLP Arsenic	7440-38-2	5.0	0.0417	0.125
TCLP Barium	7440-39-3	100	0.025	0.075
TCLP Benzene	71-43-2	0.5	0.0005	.001

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	Project Action Limit (mg/L)	Laboratory Detection Limit (mg/L)	Laboratory Reporting Limit (mg/L)
TCLP Cadmium	7440-43-9	1.0	0.00665	0.025
TCLP Carbon tetrachloride	56-23-5	0.5	0.0005	0.001
TCLP Chlordane	57-74-9	0.03		
TCLP Chlorobenzene	108-90-7	100	0.0005	0.001
TCLP Chloroform	67-66-3	6.0	0.0025	0.005
TCLP Chromium	7440-47-3	5.0	0.0127	0.05
TCLP o-Cresol	95-48-7	200	0.008681	0.0289
TCLP m-Cresol	108-39-4	200		
TCLP p-Cresol	106-44-5	200		
TCLP 2,4-D	94-75-7	10.0		
TCLP 1,4-Dichlorobenzene	106-46-7	7.5	0.018774	0.0625
TCLP 1,2-Dichloroethane	107-06-2	0.5	0.000167654	0.001
TCLP 1,1-Dichloroethylene	75-35-4	0.7	0.000410249	0.001
TCLP 2,4-Dinitrotoluene	121-14-2	0.13	0.007916	0.0264
TCLP Endrin	72-20-8	0.02		
TCLP Heptachlor	76-44-8	0.008		
TCLP Hexachlorobenzene	118-74-1	0.13	0.016933	0.0564
TCLP Hexachloro-1,3-butadiene	87-68-3	0.5	0.024611	0.082
TCLP Hexachloroethane	67-72-1	3.0	0.026593	0.0886
TCLP Lead	7439-92-1	5.0	0.0217	0.065
TCLP Lindane	58-89-9	0.4		
TCLP Mercury	7439-97-6	0.2	0.000126L	0.00042016

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	Project Action Limit (mg/L)	Laboratory Detection Limit (mg/L)	Laboratory Reporting Limit (mg/L)
TCLP Nitrobenzene	98-95-3	2.0	0.014501	0.0483
TCLP Methoxychlor	72-43-5	10.0		
TCLP Methyl Ethyl Ketone	78-93-3	200	0.002979164	0.020
TCLP Pentachlorophenol	87-86-5	100	0.014343	0.0478
TCLP Pyridine	100-86-1	5.0	0.017893	0.0596
TCLP Selenium	7782-49-2	1.0	0.0828	0.25
TCLP Silver	7440-22-4	5.0	0.0166	0.05
TCLP Tetrachloroethylene	127-18-4	0.7	0.0005	0.001
TCLP Toxaphene	8001-35-2	0.5		
TCLP Trichloroethylene	79-01-6	0.5	0.000330675	0.001
TCLP 2,4,5-Trichlorophenol	95-95-4	400	0.008422	0.028
TCLP 2,4,6-Trichlorophenol	88-06-2	2.0	0.021129	0.0704
TCLP 2,4,5-TP (Silvex)	93-72-1	1.0		
TCLP Vinyl Chloride	75-01-4	0.2	0.000175599	0.001

Notes: TCLP Project Action Limits as set by 40 CFR Part 261 Hazardous Waste Identification

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Groundwater

Analytical Method: As noted

Concentration level (if applicable):

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Volatile Organic Compounds by Method SW846-8260B				
Acetone	67-64-1	9000		
Acetonitrile	75-05-8	#N/A		
Acrolein (Propenal)	107-02-8	#N/A		
Acrylonitrile	107-13-1	#N/A		
Allyl alcohol	107-18-6	#N/A		
Allyl chloride	107-05-1	#N/A		
Benzene	71-43-2	5	0.5	1
Benzyl chloride	100-44-7	#N/A		
Bis(2-chloroethyl)sulfide	505-60-2	#N/A		
Bromoacetone	598-31-2	#N/A		
Bromochloromethane	74-97-5	#N/A	0.340288	1
Bromodichloromethane	75-27-4	0.6	0.5	1
4-Bromofluorobenzene (surr)	460-00-4	#N/A		
Bromoform	75-25-2	4.4	0.5	1

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Bromomethane	74-83-9	10	2.434473	5
n-Butanol	71-36-3	#N/A		
2-Butanone (MEK)	78-93-3	4000		
t-Butyl alcohol	75-65-0	12		
Carbon disulfide	75-15-0	1000		
Carbon tetrachloride	56-23-5	5	0.5	1
Chloral hydrate	302-17-0	#N/A		
Chlorobenzene	108-90-7	100	0.5	1
Chlorobenzene-d ₅ (IS)	3114-55-4	#N/A		
Chlorodibromomethane	124-48-1	60	0.5	1
Chloroethane	75-00-3	400	0.374502	1
2-Chloroethanol	107-07-3	#N/A		
2-Chloroethyl vinyl ether	110-75-8	#N/A		
Chloroform	67-66-3	6	2.5	5
Chloromethane	74-87-3	30	0.5	1
Chloroprene	126-99-8	#N/A		
3-Chloropropionitrile	542-76-7	#N/A		
Crotonaldehyde	4170-30-3	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
1,2-Dibromo-3-chloropropane	96-12-8	0.2	2.164361	5
1,2-Dibromoethane	106-93-4	0.05	0.17777	1
Dibromomethane	74-95-3	#N/A	0.426508	1
1,2-Dichlorobenzene	95-50-1	600	0.5	1
1,3-Dichlorobenzene	541-73-1	600	0.5	1
1,4-Dichlorobenzene	106-46-7	75	0.5	1
1,4-Dichlorobenzene-d ₄ (IS)	3855-82-1	#N/A		
cis-1,4-Dichloro-2-butene	1476-11-5	#N/A		
trans-1,4-Dichloro-2-butene	110-57-6	#N/A		
Dichlorodifluoromethane	75-71-8	1000	0.224151	1
1,1-Dichloroethane	75-34-3	850	0.241524	1
1,2-Dichloroethane	107-06-2	5	0.16808	1
1,2-Dichloroethane-d ₄ (surr)	17060-06-0	#N/A		
1,1-Dichloroethene	75-35-4	7	0.410249	1
trans-1,2-Dichloroethene	156-60-5	100	0.256577	1
1,2-Dichloropropane	78-87-5	5	0.233076	1
1,3-Dichloro-2-propanol	96-23-1	#N/A		
cis-1,3-Dichloropropene	10061-01-5	#N/A	0.5	1

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
trans-1,3-Dichloropropene	10061-02-6	#N/A	0.229606	1
1,2,3,4-Diepoxybutane	1464-53-5	#N/A		
Diethyl ether	60-29-7	1000		
1,4-Difluorobenzene (IS)	540-36-3	#N/A		
1,4-Dioxane	123-91-1	3		
Epichlorohydrin	106-89-8	#N/A		
Ethanol	64-17-5	#N/A		
Ethyl acetate	141-78-6	#N/A		
Ethylbenzene	100-41-4	700	0.5	1
Ethylene oxide	75-21-8	#N/A		
Ethyl methacrylate	97-63-2	#N/A		
Fluorobenzene (IS)	462-06-6	#N/A		
Hexachlorobutadiene	87-68-3	#N/A	2.105642	5
Hexachloroethane	67-72-1	#N/A		
2-Hexanone	591-78-6	#N/A		
2-Hydroxypropionitrile	78-97-7	#N/A		
Iodomethane	74-88-4	#N/A		
Isobutyl alcohol	78-83-1	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Isopropylbenzene	98-82-8	#N/A	0.143317	1
Malononitrile	109-77-3	#N/A		
Methacrylonitrile	126-98-7	#N/A		
Methanol	67-56-1	5000		
Methylene chloride	75-09-2	5	0.23258	1
Methyl methacrylate	80-62-6	#N/A		
4-Methyl-2-pentanone (MIBK)	108-10-1	500		
Naphthalene	91-20-3	100	2.5	5
Nitrobenzene	98-95-3	#N/A		
2-Nitropropane	79-46-9	#N/A		
N-Nitroso-di-n-butylamine	924-16-3	#N/A		
Paraldehyde	123-63-7	#N/A		
Pentachloroethane	76-01-7	#N/A		
2-Pentanone	107-87-9	#N/A		
2-Picoline	109-06-8	#N/A		
1-Propanol	71-23-8	#N/A		
2-Propanol	67-63-0	#N/A		
Propargyl alcohol	107-19-7	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
f3-Propiolactone	57-57-8	#N/A		
Propionitrile (ethyl cyanide)	107-12-0	#N/A		
n-Propylamine	107-10-8	#N/A		
Pyridine	110-86-1	10		
Styrene	100-42-5	100	0.5	1
1,1,1,2-Tetrachloroethane	630-20-6	70	0.180582	1
1,1,2,2-Tetrachloroethane	79-34-5	0.2	0.249331	1
Tetrachloroethene	127-18-4	5	0.5	1
Toluene	108-88-3	800	0.5	1
Toluene-d8 (surr)	2037-26-5	#N/A		
o-Toluidine	95-53-4	#N/A		
1,2,4-Trichlorobenzene	120-82-1	70	2.209495	5
1,1,1-Trichloroethane	71-55-6	200	0.5	1
1,1,2-Trichloroethane	79-00-5	5	0.197393	1
Trichloroethene	79-01-6	5	0.330675	1
Trichlorofluoromethane	75-69-4	3490	0.184954	1
1,2,3-Trichloropropane	96-18-4	60	0.5	1
Vinyl acetate	108-05-4	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Vinyl chloride	75-01-4	0.2	0.175599	1
o-Xylene	95-47-6	#N/A	0.5	1
m-Xylene	108-38-3	#N/A		
p-Xylene	106-42-3	#N/A		
1,3,5-Trimethylbenzene	108-68-8	#N/A	0.5	1
1,2,4-Trimethylbenzene	95-63-6	#N/A	0.5	1

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Groundwater

Analytical Method: As noted

Concentration level (if applicable):

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Semi-volatile Organic Compounds by Method SW846-8270C (PAHs by 8270 SIM)				
Acenaphthene	83-32-9	#N/A	0.00607	0.0303
Acenaphthylene	208-96-8	#N/A	0.00498	0.0249
Acetophenone	98-86-2	#N/A		
2-Acetylaminofluorene	53-96-3	#N/A		
1-Acetyl-2-thiourea	591-08-2	#N/A		
2-Aminoanthraquinone	117-79-3	#N/A		
Aminoazobenzene	60-09-3	#N/A		
4-Aminobiphenyl	92-67-1	#N/A		
3-Amino-9-ethylcarbazole	132-32-1	#N/A		
Anilazine	101-05-3	#N/A		
Aniline	62-53-3	#N/A		
o-Anisidine	90-04-0	#N/A		
Anthracene	120-12-7	3000	0.01045	0.0523
Aramite	140-57-8	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Azinphos-methyl	86-50-0	#N/A		
Barban	101-27-9	#N/A		
Benzidine	92-87-5	#N/A		
Benzoic Acid	65-85-0	#N/A		
Benz(a)anthracene	56-55-3	#N/A	0.00755	0.0378
Benzo(b)fluoranthene	205-99-2	0.2	0.00574	0.0287
Benzo(k)fluoranthene	207-08-9	#N/A	0.00755	0.0377
Benzo(g,h,i)perylene	191-24-2	#N/A	0.00678	0.0339
Benzo(a)pyrene	50-32-8	0.2	0.01053	0.0526
Benzo(e)pyrene	192-97-2	#N/A		
p-Benzoquinone	106-51-4	#N/A		
Benzyl alcohol	100-51-6	#N/A		
Bis(2-chloroethoxy)methane	111-91-1	#N/A	0.9963	3.3209
Bis(2-chloroethyl) ether	111-44-4	#N/A	1.5816	5.2721
Bis(2-chloroisopropyl) ether	108-60-1	#N/A		
Bis(2-ethylhexyl) phthalate	117-81-7	6	0.6932	2.3108
4-Bromophenyl phenyl ether	101-55-3	#N/A	1.9722	6.5739
Bromoxynil	1689-84-5	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Butyl benzyl phthalate	85-68-7	#N/A	0.7735	2.5782
Captafol	2425-06-1	#N/A		
Captan	133-06-2	#N/A		
Carbaryl	63-25-2	40		
Carbofuran	1563-66-2	40		
Carbophenothion	786-19-6	#N/A		
Chlordane (NOS)	57-74-9	#N/A		
Chlorfenvinphos	470-90-6	#N/A		
4-Chloroaniline	106-47-8	#N/A	1.097	3.6565
Chlorobenzilate	510-15-6	#N/A		
5-Chloro-2-methylaniline	95-79-4	#N/A		
4-Chloro-3-methylphenol	59-50-7	#N/A	1.6878	5.6259
3-(Chloromethyl)pyridine hydrochloride	6959-48-4	#N/A		
1-Chloronaphthalene	90-13-1	#N/A		
2-Chloronaphthalene	91-58-7	#N/A	1.6457	5.4856
2-Chlorophenol	95-57-8	#N/A	1.1566	3.8555
4-Chloro-1,2-phenylenediamine	95-83-0	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
4-Chloro-1,3-phenylenediamine	5131-60-2	#N/A		
<u>4-Chlorophenyl phenyl ether</u>	7005-72-3	#N/A	0.8194	2.7315
<u>Chrysene</u>	218-01-9	0.2	0.01305	0.0526
<u>Coumaphos</u>	56-72-4	#N/A		
4-Chloro-1,3-phenylenediamine	5131-60-2	#N/A		
<u>4-Chlorophenyl phenyl ether</u>	7005-72-3	#N/A	0.8194	2.7315
<u>Chrysene</u>	218-01-9	0.2	0.01305	0.0526
<u>Coumaphos</u>	56-72-4	#N/A		
<u>p-Cresidine</u>	120-71-8	#N/A		
<u>Crotoxyphos</u>	7700-17-6	#N/A		
<u>2-Cyclohexyl-4,6-dinitro-phenol</u>	131-89-5	#N/A		
<u>Demeton-O</u>	298-03-3	#N/A		
<u>Demeton-S</u>	126-75-0	#N/A		
<u>Diallate (cis or trans)</u>	2303-16-4	#N/A		
<u>2,4-Diaminotoluene</u>	95-80-7	#N/A		
<u>Dibenz(a,j)acridine</u>	224-42-0	#N/A		
<u>Dibenz(a,h)anthracene</u>	53-70-3	#N/A	0.01002	0.0501
<u>Dibenzofuran</u>	132-64-9	#N/A	0.7685	2.5618

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
<u>Dibenzo(a,e)pyrene</u>	192-65-4	#N/A		
<u>1,2-Dibromo-3-chloropropane</u>	96-12-8	0.2		
<u>Di-n-butyl phthalate</u>	84-74-2	1000	2.5639	8.5462
<u>Dichlone</u>	117-80-6	#N/A		
<u>1,2-Dichlorobenzene</u>	95-50-1	600	1.9296	6.432
<u>1,3-Dichlorobenzene</u>	541-73-1	600	1.883	6.2766
<u>1,4-Dichlorobenzene</u>	106-46-7	75	1.8774	6.2582
<u>3,3'-Dichlorobenzidine</u>	91-94-1	#N/A	0.9054	3.0179
<u>2,4-Dichlorophenol</u>	120-83-2	#N/A	1.3667	4.5555
<u>2,6-Dichlorophenol</u>	87-65-0	#N/A		
<u>Dichlorovos</u>	62-73-7	#N/A		
<u>Dicrotophos</u>	141-66-2	#N/A		
<u>Diethyl phthalate</u>	84-66-2	#N/A	1.0824	3.6079
<u>Diethylstilbestrol</u>	56-53-1	#N/A		
<u>Diethyl sulfate</u>	64-67-5	#N/A		
<u>Dimethoate</u>	60-51-5	2		
<u>3,3'-Dimethoxybenzidine</u>	119-90-4	#N/A		
<u>Dimethylaminoazobenzene</u>	60-11-7	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
7,12-Dimethylbenz(a)- <u>anthracene</u>	57-97-6	#N/A		
3,3'-Dimethylbenzidine	119-93-7	#N/A		
α,α -Dimethylphenethylamine	122-09-8	#N/A		
2,4-Dimethylphenol	105-67-9	#N/A	1.2651	4.2168
Dimethyl phthalate	131-11-3	#N/A	1.9304	6.4347
1,2-Dinitrobenzene	528-29-0	#N/A		
1,3-Dinitrobenzene	99-65-0	#N/A		
1,4-Dinitrobenzene	100-25-4	#N/A		
4,6-Dinitro-2-methylphenol	534-52-1	#N/A		
2,4-Dinitrophenol	51-28-5	#N/A	0.7112	2.3706
2,4-Dinitrotoluene	121-14-2	0.05	0.7916	2.6385
2,6-Dinitrotoluene	606-20-2	0.05	0.6028	2.0093
Dinocap	39300-45-3	#N/A		
Dinoseb	88-85-7	7		
Diphenylamine	122-39-4	#N/A		
5,5-Diphenylhydantoin	57-41-0	#N/A		
1,2-Diphenylhydrazine	122-66-7	#N/A		
Di-n-octyl phthalate	117-84-0	#N/A	1.8923	6.3075

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Disulfoton	298-04-4	#N/A		
EPN	2104-64-5	#N/A		
Ethion	563-12-2	#N/A		
Ethyl carbamate	51-79-6	#N/A		
Ethyl methanesulfonate	62-50-0	#N/A		
Famphur	52-85-7	#N/A		
Fensulfothion	115-90-2	#N/A		
Fenthion	55-38-9	#N/A		
Fluchloralin	33245-39-5	#N/A		
Fluoranthene	206-44-0	400	0.01067	0.0533
Fluorene	86-73-7	400	0.00797	0.0399
2-Fluorobiphenyl (surr)	321-60-8	#N/A		
2-Fluorophenol (surr)	367-12-4	#N/A		
Hexachlorobenzene	118-74-1	1	1.6933	5.6442
Hexachlorobutadiene	87-68-3	#N/A		
Hexachlorocyclopentadiene	77-47-4	#N/A	0.6784	2.2613
Hexachloroethane	67-72-1	#N/A	2.6593	8.8642
Hexachlorophene	70-30-4	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Hexachloropropene	1888-71-7	#N/A		
Hexamethylphosphoramide	680-31-9	#N/A		
Hydroquinone	123-31-9	#N/A		
Indeno(1,2,3-cd)pyrene	193-39-5	#N/A	0.01764	0.0882
Isodrin	465-73-6	#N/A		
Isophorone	78-59-1	#N/A	0.7346	2.4485
Isosafrole	120-58-1	#N/A		
Kepone	143-50-0	#N/A		
Leptophos	21609-90-5	#N/A		
Malathion	121-75-5	#N/A		
Maleic anhydride	108-31-6	#N/A		
Mestranol	72-33-3	#N/A		
Methapyrilene	91-80-5	#N/A		
3-Methylcholanthrene	56-49-5	#N/A		
4,4'-Methylenebis(2-chloroaniline)	101-14-4	#N/A		
4,4'-Methylenebis(N,N-dimethyl-aniline)	101-61-1	#N/A		
Methyl methanesulfonate	66-27-3	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
2-Methylnaphthalene	91-57-6	#N/A	0.00490	0.0295
Methyl parathion	298-00-0	#N/A		
2-Methylphenol	95-48-7	#N/A	0.8681	2.8938
3-Methylphenol	108-39-4	#N/A		
4-Methylphenol	106-44-5	#N/A		
Mevinphos	7786-34-7	#N/A		
Mexacarbate	315-18-4	#N/A		
Mirex	2385-85-5	#N/A		
Monocrotophos	6923-22-4	#N/A		
Naled	300-76-5	#N/A		
Naphthalene	91-20-3	100	0.01833	0.0916
1,4-Naphthoquinone	130-15-4	#N/A		
1-Naphthylamine	134-32-7	#N/A		
2-Naphthylamine	91-59-8	#N/A		
Nicotine	54-11-5	#N/A		
5-Nitroacenaphthene	602-87-9	#N/A		
2-Nitroaniline	88-74-4	#N/A	0.7737	2.5789
3-Nitroaniline	99-09-2	#N/A	0.9696	3.2321

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
4-Nitroaniline	100-01-6	#N/A	1.8309	6.1031
5-Nitro-o-anisidine	99-59-2	#N/A		
Nitrobenzene	98-95-3	#N/A	1.4501	4.8337
4-Nitrobiphenyl	92-93-3	#N/A		
Nitrofen	1836-75-5	#N/A		
2-Nitrophenol	88-75-5	#N/A	1.1643	3.8809
4-Nitrophenol	100-02-7	#N/A	1.0477	3.4923
5-Nitro-o-toluidine	99-55-8	#N/A		
Nitroquinoline-1-oxide	56-57-5	#N/A		
N-Nitrosodi-n-butylamine	924-16-3	#N/A		
N-Nitrosodiethylamine	55-18-5	#N/A		
N-Nitrosodimethylamine	62-75-9	#N/A		
N-Nitrosomethylethylamine	10595-95-6	#N/A		
N-Nitrosodiphenylamine	86-30-6	7	3.5279	11.7595
N-Nitrosodi-n-propylamine	621-64-7	#N/A	0.9712	3.2375
N-Nitrosomorpholine	59-89-2	#N/A		
N-Nitrosopiperidine	100-75-4	#N/A		
N-Nitrosopyrrolidine	930-55-2	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Octamethyl pyrophosphoramidate	152-16-9	#N/A		
4,4'-Oxydianiline	101-80-4	#N/A		
Parathion	56-38-2	#N/A		
Pentachlorobenzene	608-93-5	#N/A		
Pentachloronitrobenzene	82-68-8	#N/A		
Pentachlorophenol	87-86-5	1	1.4343	4.781
Perylene	198-55-0	#N/A		
Phenacetin	62-44-2	#N/A		
Phenanthrene	85-01-8	#N/A	0.01379	0.0689
Phenobarbital	50-06-6	#N/A		
Phenol	108-95-2	2000	0.5995	1.9984
1,4-Phenylenediamine	106-50-3	#N/A		
Phorate	298-02-2	#N/A		
Phosalone	2310-17-0	#N/A		
Phosmet	732-11-6	#N/A		
Phosphamidon	13171-21-6	#N/A		
Phthalic anhydride	85-44-9	#N/A		
2-Picoline (2-Methylpyridine)	109-06-8	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Piperonyl sulfoxide	120-62-7	#N/A		
Pronamide	23950-58-5	#N/A		
Propylthiouracil	51-52-5	#N/A		
Pyrene	129-00-0	250	0.00765	0.0383
Resorcinol	108-46-3	#N/A		
Safrole	94-59-7	#N/A		
Strychnine	57-24-9	#N/A		
Sulfallate	95-06-7	#N/A		
Terbufos	13071-79-9	#N/A		
1,2,4,5-Tetrachlorobenzene	95-94-3	#N/A		
2,3,4,6-Tetrachlorophenol	58-90-2	#N/A		
Tetrachlorvinphos	961-11-5	#N/A		
Tetraethyl dithiopyrophosphate	3689-24-5	#N/A		
Tetraethyl pyrophosphate	107-49-3	#N/A		
Thionazine	297-97-2	#N/A		
Thiophenol (Benzenethiol)	108-98-5	#N/A		
Toluene diisocyanate	584-84-9	#N/A		
o-Toluidine	95-53-4	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
1,2,4-Trichlorobenzene	120-82-1	70	2.0347	6.7823
2,4,5-Trichlorophenol	95-95-4	#N/A	0.8422	2.8073
2,4,6-Trichlorophenol	88-06-2	#N/A	2.1129	7.0429
Trifluralin	1582-09-8	7.5		
2,4,5-Trimethylaniline	137-17-7	#N/A		
Trimethyl phosphate	512-56-1	#N/A		
1,3,5-Trinitrobenzene	99-35-4	#N/A		
Tris(2,3-dibromopropyl) phosphate	126-72-7	#N/A		

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: [Groundwater](#)

Analytical Method: 8082

Concentration level (if applicable):

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Polychlorinated Biphenyls by Method SW846-8082				
Aroclor 1016	12674-11-2	#N/A	0.25	0.5
Aroclor 1221	11104-28-2	#N/A	0.25	0.5
Aroclor 1232	11141-16-5	#N/A	0.25	0.5
Aroclor 1242	53469-21-9	#N/A	0.25	0.5
Aroclor 1248	12672-29-6	#N/A	0.25	0.5
Aroclor 1254	11097-69-1	#N/A	0.25	0.5
Aroclor 1260	11096-82-5	#N/A	0.25	0.5
Aroclor 1262	37324-23-5	#N/A	0.25	0.5
Aroclor 1268	11100-14-4	#N/A	0.25	0.5

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: Groundwater

Analytical Method: As noted

Concentration level (if applicable):

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Inorganic Compounds by Method SW846-6020A				
Aluminum	7429-90-5	200	58.700	250.00
Antimony	7440-36-0	6	0.150	1.00
Arsenic	7440-38-2	10	0.279	1.00
Barium	7440-39-3	2000	0.341	1.14
Beryllium	7440-41-7	4	0.178	1.00
Cadmium	7440-43-9	5	0.081	1.00
Calcium	7440-70-2	#N/A	69.774	250.00
Chromium	7440-47-3	100	1.0199	3.40
Cobalt	7440-48-4	40	0.085	1.00
Copper	7440-50-8	1300	1.093	3.65
Cyanide, Free	57-12-5	200	0.00679	0.023
Iron	7439-89-6	#N/A	110.551	368.50
Lead	7439-92-1	15	0.195	1.00
Magnesium	7439-95-4	#N/A	29.700	250.00

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Manganese	7439-96-5	300	2.700	9.00
Mercury (SW-846 7471B)	7439-97-6	2	0.179	0.597
Nickel	7440-02-0	100	0.402	1.34
Potassium	7440-09-7	#N/A	236.721	789.10
Selenium	7882-49-2	50	0.317	1.06
Silver	7440-22-4	50	0.101	0.50
Sodium	7440-23-5	#N/A	42.000	250.00
Thallium	7440-28-0	2	0.140	1.00
Vanadium	7440-62-2	30	0.316	1.05
Zinc	7440-66-6	#N/A	4.600	15.33

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.

Matrix: [Groundwater](#)

Analytical Method: [As noted](#)

Concentration level (if applicable):

Analyte	CAS No.	WI NR140 GW ES (ug/L)	Laboratory Detection Limit (ug/L)	Laboratory Reporting Limit (ug/L)
Geochemical Parameters				
Alkalinity, Total ((2320B)	7429-90-5	#NA	5000	10000
Nitrate-Nitrite, Total (353.2)	7440-36-0	#NA	95	250
Sulfate, Total (EPA 300.0)	14808-79-8	#NA	2000	4000

NOTE: #NA = no regulatory concentration for the indicated analyte.

Refer to Section 1.5.3 – Step 3 Decision Inputs of the Addendum to USEPA-Approved Multi-Site Quality Assurance Project Plan for discussion of reporting limits and detection limits that exceed the respective screening levels.



Attachment 2
Analytical Laboratory
Standard Operating
Procedures



Attachment 2-1
Pace Analytical Services, LLC

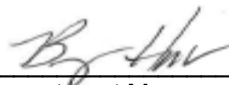


STANDARD OPERATING PROCEDURE

Reactive Cyanide and Reactive Sulfide,
SW-846 Chapters 7.3.3.2 (Cyanide) and 7.3.4.2 (Sulfide)

SOP NUMBER: PGH-I-017-6
REVIEW: David Harlin
EFFECTIVE DATE: Date of Final Signature
SUPERSEDES: PGH-I-017-5
REVIEW DATE: Upon Procedural Change

APPROVALS



Department Manager/Supervisor 08/11/17
Date



Senior Quality Manager 08/14/17
Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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1. Purpose
 - 1.1 This SOP documents the procedure to be followed for the analysis of aqueous and solid samples for Reactive Cyanide and Reactive Sulfide per SW-846 Chapters 7.3.3.2 and 7.3.4.2.
 - 1.2 Reactive Cyanide is analyzed by EPA 9014 (PGH-I-053). Reactive Sulfide is analyzed by SM4500 S₂-F-00 (PGH-I-010).
2. Scope and Application
 - 2.1 This SOP is applicable to all wastes (solid, aqueous and organic) provided that they do not form an explosive or toxic mixture when combined with acids.
 - 2.2 This method is used to determine the specific amount of hydrocyanic acid and hydrogen sulfide generated upon contact with aqueous acid and is used, in part, to determine reactivity for hazardous waste characterization.
 - 2.3 This test determines only the hydrocyanic acid (HCN) and hydrogen (H₂S) sulfide evolved at the specific test conditions and is not intended to measure forms of cyanide other than those evolvable under these test conditions.
 - 2.4 This method is used to simultaneously generate results for reactive cyanide and reactive sulfide.
 - 2.5 The current reporting limits and method detection limits (MDLs) for reactive cyanide and reactive sulfide are in the LIMS and available from the Quality department.
3. Summary of Method
 - 3.1 An aliquot of acid is added slowly to a known mass of waste in a closed system. The generated gas is swept into a scrubber containing lead acetate which captures sulfide and then to a scrubber containing sodium hydroxide (NaOH) which captures cyanide.
 - 3.2 The HCN is quantified colorimetrically according to the determinative step in the SOP for total cyanide analysis (PGH-I-053).
 - 3.3 The acetate solution binds the sulfide (if present) and forms a dark precipitate. The presence of the dark precipitate determines the need for the second step. This screening test is sufficiently sensitive to detect sulfide well below the reporting limits (RLs) of the quantitative analytical step of the test.
 - 3.4 If precipitate is observed in the lead acetate scrubber, a second sample aliquot is tested and the evolved gas is swept into a scrubber containing sodium hydroxide. The scrubber contents are analyzed titrimetrically according to the determinative step in the SOP for total sulfide (PGH-I-010).
4. Interferences
 - 4.1 Interferences for this method are undetermined.
 - 4.2 Refer to the interference sections of the determinative methods in the SOPs for total cyanide (PGH-I-053) and total sulfide (PGH-I-010).
5. Safety
 - 5.1 Testing should be performed in a hood.
 - 5.2 Use extreme caution when acidifying samples that contain large amounts of cyanide and/or sulfide because both hydrogen cyanide (HCN) and hydrogen sulfide (H₂S) are extremely toxic.

-
- 5.3 Refer to the Pace Analytical Services, LLC – Pittsburgh Chemical Hygiene Plan/Safety Manual for the specific safety requirements to be followed when working in the laboratory.
 - 5.4 The toxicity and carcinogenicity of each reagent used in this procedure has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. At a minimum, personal protective equipment must include a lab coat, gloves, and safety glasses.
 - 5.5 Analysts must be familiar with the Safety Data Sheets (SDS) for all chemicals and reagents used in this procedure, and the location of the SDS within the laboratory.
6. Definitions
- 6.1 Refer to the Glossary Section of the most recent revision of the Pace Analytical Services, LLC Quality Manual for the definitions used throughout this SOP.
 - 6.2 Cyanide bearing waste: A waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic cyanide gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
 - 6.3 Sulfide bearing waste: A waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic hydrogen sulfide gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
7. Responsibilities and Distribution
- 7.1 General Manager/Assistant General Manager (GM/AGM)
 - 7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.
 - 7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.
 - 7.2 Senior Quality Manager/Quality Manager (SQM/QM)
 - 7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.
 - 7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.
 - 7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.
 - 7.3 Department Manager/Supervisor
 - 7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs
 - 7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.
 - 7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

-
- 7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.
 - 7.4 Individual Staff
 - 7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.
 - 7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.
 - 7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.
 - 8. Sample Collection, Preservation, and Handling
 - 8.1 Sample collection: Samples suspected of containing sulfide or a combination of sulfide and cyanide should be collected with minimal aeration. The sample bottle should be filled completely and the analysis should commence as soon as possible.
 - 8.2 Preservation: Samples may be preserved with a strong base to adjust the pH to 12. However, this will cause dilution of the sample, and possibly change other physical and chemical characteristics of the waste, which may affect the rate of release of hydrocyanic acid.
 - 8.3 Shipment: Samples should be shipped in a timely manner, in a cooler.
 - 8.4 Storage: Samples should be stored in a refrigerator at $\leq 6^{\circ}\text{C}$ and in the dark.
 - 8.5 Holding Time: 28 days.
 - 9. Equipment and Supplies
 - 9.1 See Figures 1, 2, and 3 for apparatus assembly.
 - 9.2 500mL round bottom, two-neck flasks with 24/40 ground glass joints.
 - 9.3 Addition funnels with stop cocks and 24/40 ground glass joints.
 - 9.4 Nitrogen supply.
 - 9.5 Nitrogen manifold suitable for regulating the nitrogen flow to 60mL/min.
 - 9.6 Glass adapters for connecting tubing to each flask.
 - 9.7 Flexible plastic tubing (Nalgene lab grade 5/16" ID or equivalent).
 - 9.8 Cork rings.
 - 9.9 Stir plates.
 - 9.10 100mL, 250mL, and 1L Class A graduated cylinders and volumetric flask.
 - 9.11 Two-holed stoppers for graduated cylinders with inlet extension.
 - 9.12 Top loading balance capable of measuring 0.01g.
 - 9.13 Magnetic stir bars.
 - 9.14 Timer with alarm.
 - 9.15 Volumetric flask, 1L glass Class A.
 - 9.16 Class A, calibrated pipette and pipette tips.
 - 10. Reagents and Standards

-
- 10.1 Reagent grade deionized (DI) water.
 - 10.2 1.0N Sulfuric acid, certified (purchased from Fisher Scientific, or equivalent).
 - 10.3 0.01N Sulfuric acid working solution: Measure 2mL 1.0 N sulfuric acid into a 250ml Class A graduated cylinder and dilute to 200mL with DI water. Mix well.
 - 10.4 10N Sodium hydroxide solution, certified (purchased from Fisher scientific, or equivalent).
 - 10.5 0.25N Sodium hydroxide solution: Measure 25mL NaOH into a 1L volumetric flask and dilute to volume with DI water. Mix water. If making individually, add 1.25ml of 10N NaOH to 50ml of DI water.
 - 10.6 Lead acetate solution: Place 30.42g lead acetate in a 1L volumetric flask. Dilute to the volume with DI water, and adjust the pH to 4.5 with glacial acetic acid. Mix well. This solution must be discarded after 6 months.
 - 10.7 6N HCl: To a 1L volumetric add 500mL of DI water and then slowly add 500mL of concentrated HCl. Mix well.
 - 10.8 Stock Cyanide Standard Solution (1000mg/L): This is purchased commercially. (e.g., Spex Certiprep Catalog # RSCN9-2Y, or equivalent) For commercially prepared stock, refer to manufacturer's expiration date.
 - 10.9 Stock Sulfide Standard Solution (1000mg/L): This is purchased commercially. (e.g. Aqua Solutions #8975, or equivalent). For commercially prepared stock, refer to manufacturer's expiration date.
 - 10.10 Glass beads, Fisher brand or equivalent.
 - 11. Calibration
 - 11.1 Not Applicable
 - 12. Procedure
 - 12.1 On a top loading balance, tare a cork ring and a 500mL flask. Add approximately 10g of the sample (aqueous or solid) to the flask and record this weight on the electronic bench sheet. Record the sample number on the flask. Add a rounded magnetic stir bar to the flask.
 - 12.1.1 Method/Reagent Blank prep: add approximately 10g of glass beads to the flask. Record information and add stir bar as stated in 12.1.
 - 12.1.2 Cyanide LCS prep: Pipette 1mL of Stock Cyanide Standard Solution into the flask and add DI up to approximately 10g. Add 10 g of glass beads to the flask as well. Record information and add stir bar as stated in 12.1.
 - 12.1.3 Sulfide LCS prep: Pipette 2mL of Stock Sulfide Standard Solution in the flask and add DI up to approximately 10g. Add 10 g of glass beads to the flask as well. Record information and add stir bar as stated in 12.1.
 - 12.1.4 The LCS's must be prepared in separate flasks.
 - 12.2 Place the cork ring and flask with sample on a stir plate in the hood. Place 200mL of 0.01N sulfuric acid into the addition funnel and position the funnel into one of the flask necks. Place one glass adapter into the other flask neck and one glass adapter into the top of the addition funnel (See Figure 1.)
 - 12.3 Via flexible tubing, connect:
 - 12.3.1 The nitrogen gas nozzle (from the nitrogen manifold) to the top of addition funnel;

-
- 12.3.2 The glass adapter on flask to scrubber #1 inlet tube (Figures 1 and 2) and,
- 12.3.3 The outlet from scrubber #1 to the inlet of scrubber #2 (figure 2).
- 12.4 In scrubber #1 (a 250mL plastic graduated cylinder), add 40mL lead acetate solution and dilute to approximately 160mL with DI water. Place the rubber stopper onto the graduated cylinder so that the inlet extension is submerged in the lead acetate solution (figure 2). The lead acetate solution binds the sulfide present in the sample. Sulfide is a known interferent to colorimetric quantitation of cyanide.
- 12.5 In scrubber #2 (a second 250mL graduated cylinder), add 50mL 0.25N NaOH solution. Place the rubber stopper with an elbow tube and inlet extension into the cylinder so the extension is submerged into the NaOH solution. Allow the other elbow tube to remain open to vent nitrogen gas.
- 12.6 Turn nitrogen gas on to a flow rate of 60mL/min.
- 12.7 Turn on the stir plate. NOTE: the stirring should not be so fast as to create a vortex when the acid is added.
- 12.8 With nitrogen flowing, slowly introduce all 200mL of the 0.01N H₂SO₄ from the addition funnel into the flask. At this point, set the timer for 30 minutes.
- 12.9 After 30 minutes, turn off the nitrogen gas and stir plate and disconnect the scrubbers. Place the solution into an appropriately labeled plastic container. Determine the amount of cyanide colorimetrically according to the determinative step in the SOP for total cyanide (PGH-I-053).
- 12.10 Check the contents of scrubber #1 for precipitate. If no precipitate is observed, record "No" on the bench sheet. This result is reported as "less than" the detection limit. If precipitate is observed, proceed to 12.11.
- 12.11 Repeat the procedure from step 12.1 through 12.9 using only one scrubber containing 5mL of 10N NaOH diluted to 200mL with DI water. (See Figure 3).
- 12.12 The trapping solution must be brought to a pH of 2 before proceeding. Titrate a small aliquot of the trapping solution to a pH of 2 end point with 6N HCL and calculate the amount of HCl needed to acidify the entire scrubber solution. Combine the small acidified aliquot with the remainder of the acidified scrubber solution.
- 12.13 Determine the amount of sulfide titrimetrically according to the determinative step in the SOP for total sulfide (PGH-I-010).
13. Calculations
- 13.1 See the SOPs for total cyanide (PGH-I-053) and total sulfide analysis (PGH-I-010) for the calculations pertaining to the determinative steps.
14. Quality Control
- 14.1 Analyze one reagent blank per analysis batch up to 20 samples. The reagent blank consists of glass beads and is analyzed in the same manner as analytical samples.
- 14.1.1 The Method/Reagent Blank must be free of detectable cyanide and sulfide to ensure no cross contamination (Result < Reporting Limit).
- 14.2 Analyze one Laboratory Control Sample (LCS) per analysis batch up to 20 samples for each Cyanide and Sulfide.
- 14.2.1 LCS for reactive sulfide must have a % Recovery between 0-52%.

- 14.2.2 LCS for reactive cyanide must have a % Recovery between 0-8%.
- 14.3 Analyze one duplicate (DUP) sample per analysis batch up to 20 samples.
 - 14.3.1 Duplicate sample recoveries must agree within 20% RPD
- 14.4 Corrective Actions for Out-Of-Control Data
 - 14.4.1 Method Blank (Reagent Blank) (MB/RB): Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.
 - 14.4.2 Laboratory Control Sample (LCS): Reanalyze the LCS once. If the problem persists, reprep and reanalyze the batch with the associated sample if LCS is outside acceptance limits.
 - 14.4.2.1 If LCS %Recovery is outside the QC limits high and samples are non-detect, the sample data may be reported with appropriate data qualifiers.
 - 14.4.3 Duplicate (DUP): DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.
 - 14.4.4 If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.
- 14.5 Any data that are considered out-of-control (suspect) or unacceptable will be appropriately flagged as such and qualified in the final report.
- 15. Method Performance
 - 15.1 If contamination is found in the reagent blank or duplicate samples are outside of acceptable limits, check the apparatus for cleanliness, leaks in the system, or contamination of the glass beads or reagents.
 - 15.2 Each analyst to perform this analysis must read and understand this procedure with written documentation maintained in their training file.
 - 15.3 An initial demonstration of capability (IDOC) must be performed. A record of the IDOC will be maintained on file in QA.
 - 15.4 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).
- 16. Pollution Prevention and Waste Management
 - 16.1 Spills are promptly cleaned up using paper towels, sorbent materials or spill kits, depending on the amount of and specific substance spilled. Specific protocols for handling spills are found in Pace Analytical's Chemical Hygiene Plan and in the SDSs.
 - 16.2 Procedures for handling waste generated during this analysis are addressed in PGH-C-017 Waste Management and Disposal.
 - 16.3 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time.
 - 16.4 The company wide Chemical Hygiene and Safety manual contains additional information on pollution prevention.

-
- 16.5 If a high level of reactive cyanide or sulfide is found, the sample must be segregated and disposed of in accordance with the laboratory's hazardous waste disposal policy.
17. References
- 17.1 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).
- 17.2 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.
- 17.3 Pace Analytical Services, LLC - Pittsburgh Laboratory Quality Assurance Manual, current version.
- 17.4 Test Method to Determine Hydrogen Cyanide Released from Wastes, USEPA, SW-846 Chapter 7.3.3.2, Interim Guidance For Reactive Cyanide, Revision 3, 1996.
- 17.5 Test Method to Determine Hydrogen Sulfide Released from Wastes, USEPA, SW-846 Chapter 7.3.4.2, Interim Guidance For Reactive Sulfide, Revision 3, 1996.
- 17.6 U.S. Environmental Protection Agency, Methods for the determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, EPA Method 335.4, Revision 1. August 1993.
- 17.7 USEPA, SW-846 III Ed., Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, Method 9014, September 1986.
- 17.8 Standard Method SM4500-S2-F.
- 17.9 SOP PGH-C-001, Sample Management, current version.
- 17.10 SOP PGH-Q-045, Control Charts, current version.
- 17.11 SOP PGH-C-032, Support Equipment, current version.
- 17.12 SOP PGH-Q-035, MDL/LOD/LOQ, current version.
- 17.13 SOP PGH-C-037, Standard and Reagent Traceability, current version.
- 17.14 SOP PGH-Q-038, Laboratory Equipment, current version.
- 17.15 SOP PGH-Q-039, Corrective Action, current version.
- 17.16 SOP PGH-Q-040, Internal and External Audits, current version.
- 17.17 SOP S-ALL-Q-020, Training, current version.
- 17.18 SOP S-ALL-Q-028, Lab Track, current version.
- 17.19 SOP PGH-C-027, DI Water, current version.
18. Tables, Diagrams, Flowcharts, Appendices, etc.
- 18.1 Figure 1: Distillation setup.
- 18.2 Figure 2: Cyanide (Analysis)/Sulfide (Screening) Scrubber setup.
- 18.3 Figure 3: Sulfide (Analysis) Scrubber setup.
- 18.4 Attachment 1 - Example Reactive Cyanide/Sulfide Prep Benchsheet.
19. Method Modifications
- 19.1 Two scrubbers are described in this SOP (Section 12.4). A second scrubber is not included in the reference method.

- 19.2 The concentration and volume of the NaOH solution used in the sulfate scrubber (Section 15.3) differs from the reference method.

20. Revisions

Document No.	Reason for Change	Date
PGH-I-017-2	<ol style="list-style-type: none"> Cover Page: Added Department Supervisor/Manager signature line, added Periodic Review signature lines. Updated revision numbers. Added Table of Contents to this document. Section 17: Added TNI Reference. Section 19: Added Revision History Section. 	29May2012
PGH-I-017-3	<ol style="list-style-type: none"> Cover and section 1.1: Added specific method references. Section 2.5: Added boilerplate language regarding the RLs and MDLs. Sections 3.2, 3.4, 4.2, 12.9, 12.12, 13.1: Added specific SOP references. Section 8.4: Added holding time of 28 days to match LIMS. Section 12.6: Changed to 60mL/min to match method. Section 14.1.1: Specified blank evaluation level. Removed Section 17.6: 376.1 does not exist any longer. Section 17.7: Replaced method 9034 with applicable Standard Method. Method 9034 is not performed currently by the lab. General: made editorial corrections. Added Section 19: Method Modifications. Reworded Section 10.7. Document Reformatted. Removed CN spread sheet as attachment No 2 Updated example spread sheet for sulfide as attachment No 2 	31May2014
PGH-I-017-4	<ol style="list-style-type: none"> Added Section 12.13 to acidify the sample prior to titration added 6N HCl to Section 10.8 	9-19-14
PGH-I-017-5	<ol style="list-style-type: none"> Updated 1.1 to reflect both solid and aqueous samples. Added analysis methods for reactive cyanide and sulfide as section 1.2. Updated Table of Contents. Removed example bench sheets. Added 9.16: pipette and pipette tips. Added 10.9 and 10.10 for LCS standards. Expanded 12.1 to include prep of blank, Cyanide LCS and Sulfide LCS. Corrected 12.3.3 to reference the correct diagram. Updated for electronic bench sheet throughout. Moved 12.13 to before the step directing the analyst to determine sulfide content iodometrically. Removed spreadsheet reference from 13.1. Updated section 14 to include LCS and its corrective action. Updated sections 15 and 16 to match language used in other SOPs. Updated storage temp to $\leq 6^{\circ}\text{C}$. Added additional references to Section 17. 	27Dec2016
PGH-I-017-6	<ol style="list-style-type: none"> Updated Section 9.10 added class A graduated cylinder and volumetric flask. Updated section 9.14 Timer with alarm. 	14Aug2017

Document No.	Reason for Change	Date
	<ul style="list-style-type: none">3. Removed section 10.3.4. Updated section 10.3 and 10.5.5. Updated section 12.1.2, add 10g of glass beads to the flask for cyanide.6. Updated section 12.1.3, add 10g of glass beads to the flask for sulfide.7. Updated section 12.11;... 10N NaOH diluted to 200mL with DI water.8. Added Example Reactive Cyanide/Sulfide Prep Benchsheet.	

Figure No. 1 - Distillation Setup

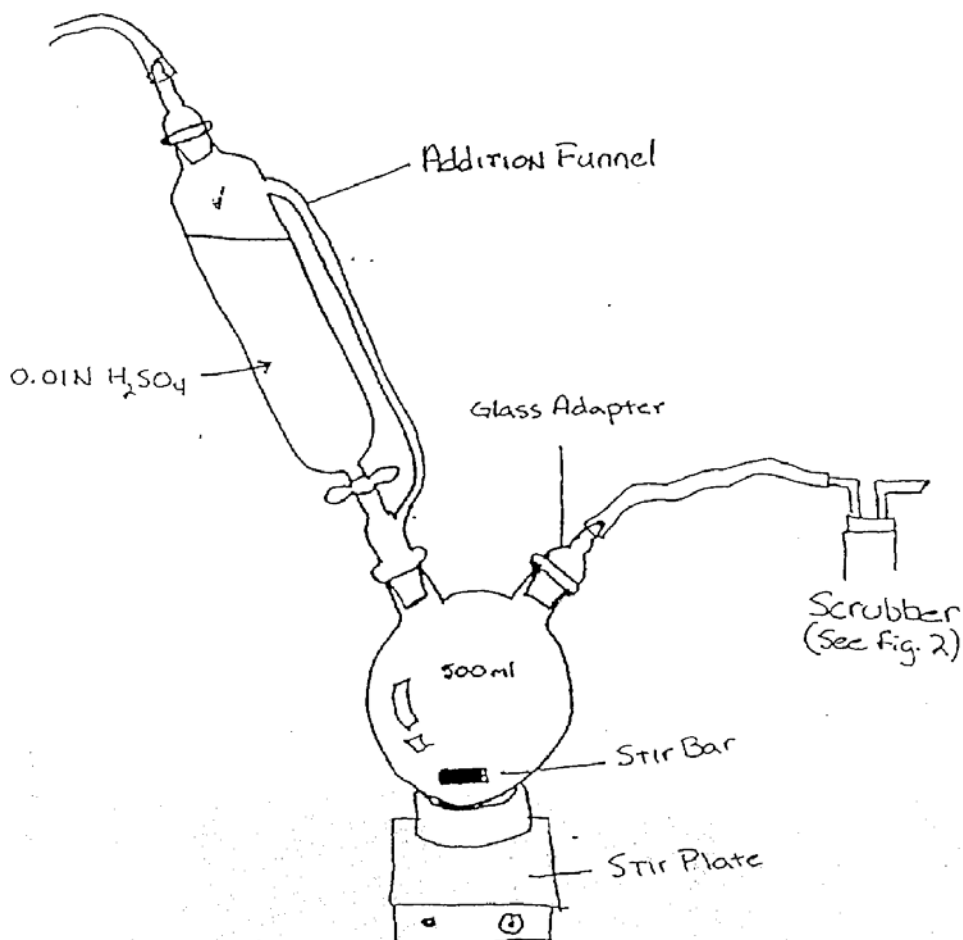


Figure No. 2 - Cyanide (Analysis)/Sulfide (Screening) Scrubber Setup

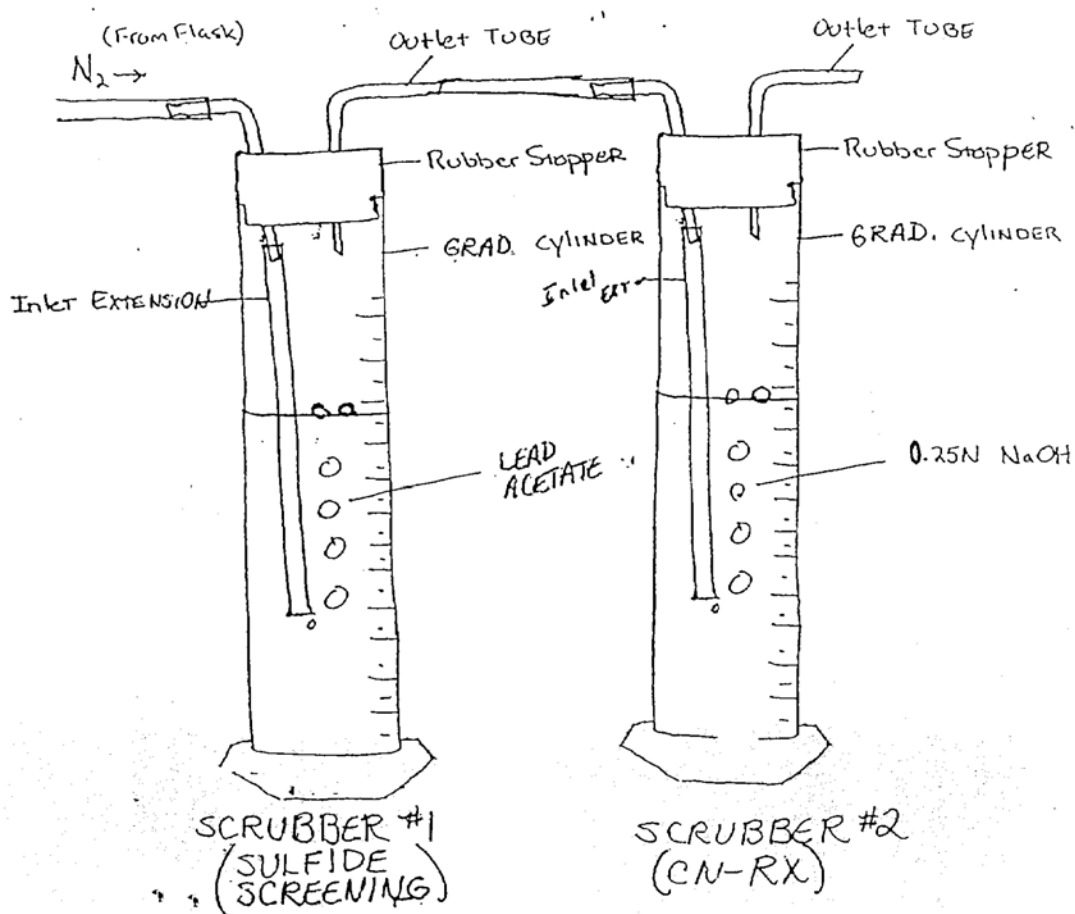
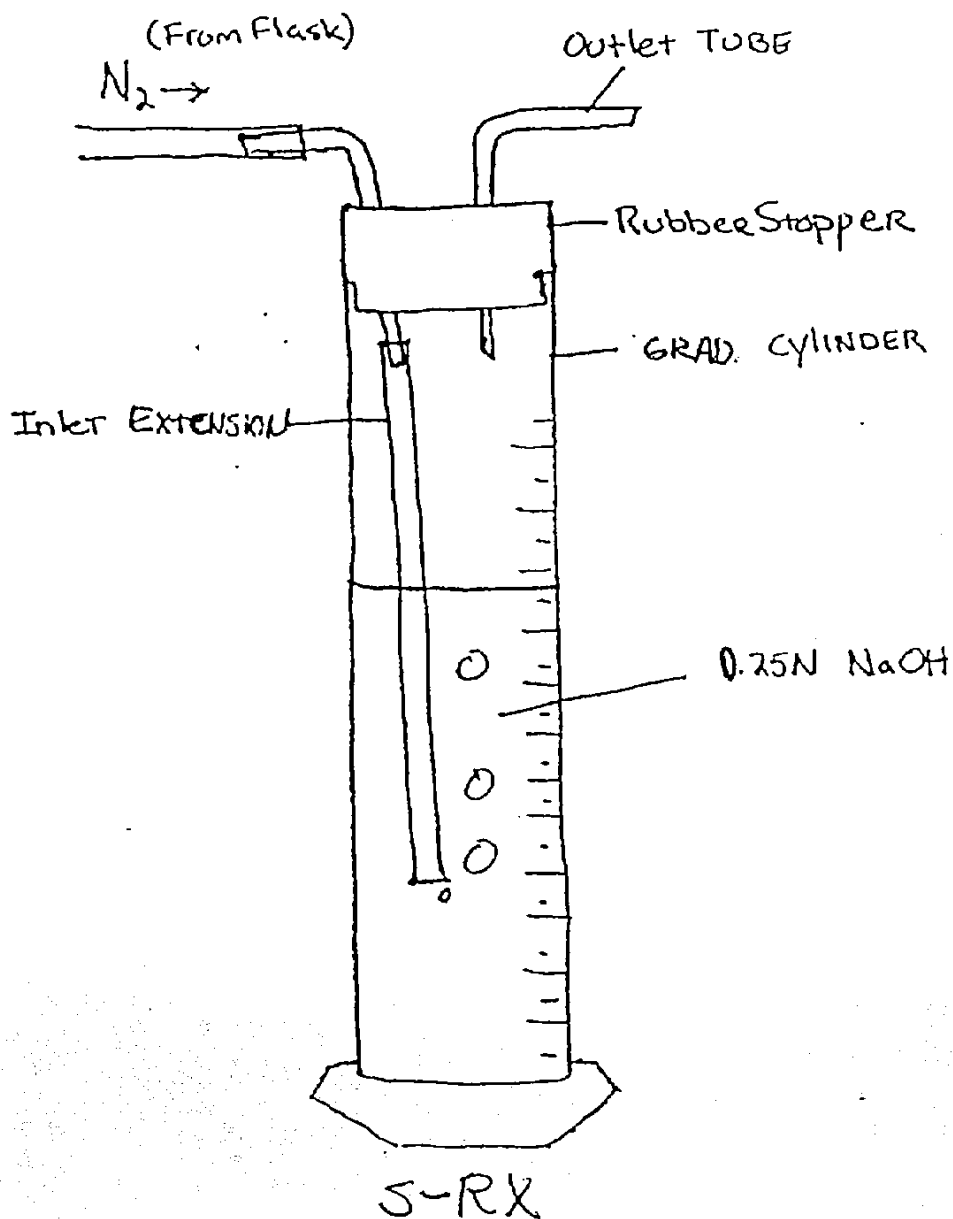


Figure No. 3 - Sulfide (Analysis) Scrubber Setup



Attachment 1 - Example Reactive Cyanide/Sulfide Prep Benchsheet



Prep Log Report

Batch Information: WET 267996/267997 P Rx

Template Version: F-PA-I-031-Rev.00 (05Nov2016)

Prep Method	SW-846 7.3.4.2	Extracted By	PAS	Extracted Date/Time	08/10/2017 13:56:16:306	Instrument	30BA14
Date/Time On	08/10/2017 14:46:36:287	Date/Time Off	08/10/2017 17:49:18:209	Lead Acetate Solution	2660	Sulfuric Acid	28065
10N Sodium Hydroxide	29775	Reviewed By	EHW	Reviewed By Date	08/11/2017 09:06	Batch Notes	

Sample Information:

QC Rule	Sample Type	Lab Sample ID	Initial Weight (g)	Final Volume (mL)	Precipitate Present	Sample Notes	CN-SPK (mL)	S-SPK (mL)
734S S2_P	BLANK	1318888	10.02	200	No			
733C S_P	BLANK	1318891	10.02	50	No			
734S S2_P	LCS	1318889	10	200	No			26333 (2)
733C S_P	LCS	1318892	10.03	50	No		24827 (1)	
734S S2_P	PS	30226596001	10.01	200	No			
733C S_P	PS	30226596001	10.01	50	No			
734S S2_P	PS	30226210001	10.01	200	No			
733C S_P	PS	30226210001	10.01	50	No			
734S S2_P	PS	30226210002	10	200	No			
733C S_P	PS	30226210002	10	50	No			
734S S2_P	PS	30226461001	10	200	No			
733C S_P	PS	30226461001	10	50	No			
734S S2_P	PS	30226462001	10.04	200	No			
733C S_P	PS	30226462001	10.04	50	No			
734S S2_P	PS	30226463001	10.06	200	No			
733C S_P	PS	30226463001	10.06	50	No			
734S S2_P	PS	461803001	10	200	Yes			
733C S_P	PS	461803001	10	50	No			
734S S2_P	PS	30226526001	10.06	200	No			
733C S_P	PS	30226526001	10.06	50	No			
734S S2_P	PS	30226692001	10.01	200	No			
733C S_P	PS	30226692001	10.01	50	No			



Prep Log Report

QC Rule	Sample Type	Lab Sample ID	Initial Weight (g)	Final Volume (mL)	Precipitate Present	Sample Notes	CN-SPK (mL)	S-SPK (mL)
734S S2_P	DUP	1318890	10.04	200	No			
733C S_P	DUP	1318893	10.04	50	No			
734S S2_P	PS	30226611001	10.06	200	No			
734S S2_P	PS	30226616001	10.07	200	No			
734S S2_P	PS	30226746001	10	200	No			

Standard Notes:

24827: WET Rx CN Spike Sol'n/2nd CN stock

26333: WET Rx Sulfide Spike Sol'n / 1000 ppm std



Pace Analytical Services, LLC. – Green Bay WI
1241 Bellevue Street Suite 9
Green Bay, WI 54303
Phone: 920 469 2436
Fax: 920 469 8827

STANDARD OPERATING PROCEDURE

SAMPLE MANAGEMENT

Reference Methods: N/A

Local SOP Number:	S-GB-C-010-Rev.08
Effective Date:	Date of Final Signature
Supersedes:	S-GB-C-010-Rev.07
SOP Template Number:	SOT-ALL-C-001-rev.06

APPROVALS

 Nils Melberg, Laboratory General Manager	<u>04/27/17</u> Date
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 Kate Verbeten, Laboratory Quality Manager	<u>4/3/2017</u> Date
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 Alea Her, Client Services Supervisor	<u>4/27/17</u> Date
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PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to outline the procedures involved with the receipt, login, storage, and disposal of samples received by Pace Analytical Services, LLC.

2. Summary of Method

2.1. Samples are delivered to the laboratory via several delivery mechanisms. Samples received are checked for adherence to the Sample Acceptance Policy (see Attachment I) with any discrepancies noted.

Discrepancies are communicated to the client if necessary for their acknowledgement and decision making.

2.2. The Laboratory Information Management System (LIMS) assigns all samples with a unique sample number and manages the analyses assigned to each sample.

2.3. Samples are labeled with the appropriate information and staged in refrigerated sample storage coolers if temperature preservation is required or possibly stored on open shelves for samples not requiring sub-ambient temperature preservation. Samples will remain under these conditions until prepared and/or analyzed. Samples received under United States Department of Agriculture (USDA) protocols need to be stored separately (please refer to the lab's Regulated Soils SOP, if applicable).

2.4. Samples and associated sub-samples (digestates, extracts, etc.), are maintained for a minimum of 45 days from receipt of samples unless otherwise requested by the client or other regulatory agency.

2.5. Samples are disposed of in accordance with local laboratory regulatory requirements, waste handling procedures, and any USDA regulated soil requirements.

3. Scope and Application

3.1. **Personnel:** The policies and procedures contained in this SOP apply to all personnel involved in the receipt, login, storage, and disposal of samples.

3.2. The Sample Acceptance Policy (Attachment I) contains the guidelines for acceptable sample conditions. Any deviation from these guidelines requires detailed documentation within the report, usually as a footnote, or on the chain-of-custody (COC), or Sample Condition Upon Receipt (SCUR) form and may require client contact.

3.3. **Parameters:** Not applicable to this SOP.

4. Applicable Matrices

4.1. Refer to Table 8.1 in this SOP for the applicable matrices.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Samples may be prone to cross contamination from others within the same delivery group or from other client projects. The sample receiving personnel must make every effort to minimize cross- contamination.

6.2. Preservation checks are one of the most likely situations where cross-contamination may occur. Materials used in the process must be specific to each sample and may not be used for multiple samples or multiple containers of the same sample.

6.3. Samples are stored under specific conditions and in specific locations, typically per the requirements of the analytical method. However, consideration must be given to samples that are uniquely different from others. Samples that are anticipated to be severely contaminated must be segregated from others in anticipation that the high levels of contaminants may cross-contaminate others in close proximity. USDA samples must also be distinctly segregated for storage.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Acceptable sample preservation, containers, and hold times can be referenced in the Bottle and Preservation Table, available within the Pace Quality Assurance Manual, or as a separate document. Samples are stored separately from all standards and reagents and any known highly contaminated samples.

7.2. **NOTE:** To avoid contamination, no food or drink products can be located near the areas where samples are unpacked, labeled, or staged.

7.3. Sample Storage – See Section 12.3 for general storage guidelines.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Chain-of-Custody (COC):** a form used to record the field identification of samples collected, analyses requested, date and time of collection, sample preservation used, and traceability of samples from time of collection until delivery to the laboratory. This is a legal document.

8.3. **Laboratory Information Management System (LIMS):** a computer system used to manage the flow and traceability of environmental samples and associated data within the laboratory.

8.4. **Matrix:** the bulk characteristics of a sample. See Table 8.1 below.

8.5. **Safety Data Sheet (SDS):** contains information on chemicals used in the laboratory.

8.6. **Sample Custody:** a sample is considered to be in someone's custody if:

8.6.1. It is in one's physical possession;

8.6.2. It is in someone's view, after being in someone's physical possession;

8.6.3. It is kept in a secured area, restricted to authorized personnel only.

8.7. **Sample Condition Upon Receipt (SCUR) form:** a form used to record the condition of samples received in the laboratory.

8.8. **Sample Receipt Form (SRF):** form generated by LIMS system after a project is logged in. Contains sample and project information.

8.9. **UN Number** - identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international and domestic transportation. These shipping names along with the identification numbers are located in the Federal Register (49CFR172.101).

Table 8.1

NELAC/TNI defined matrix	Corresponding EPIC Pro matrices
Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device.	Air (AR)
Aqueous: any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.	Water (WT)
Biological tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.	Tissue (TS) or Tissue Dry (TD)
Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.	Oil (OL) or Other (OT)
Drinking Water: any aqueous sample that has been designated a potable or potentially potable water source.	Drinking Water (DW)
Non-aqueous liquid: any organic liquid with < 15% settleable solids.	Other (OT)
Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.	Water (WT)- not assigned as a separate matrix.
Solids: includes soils, sediments, sludges and other matrices with > 15% settleable solids.	Solid (SL)
(No corresponding matrix to wipes; wipes would be included in with solids)	Wipe (WP) or Swab (SW)

9. Equipment and Supplies (Including Computer Hardware and Software)

Table 9.1

Equipment/Supplies	Description	Vendor/Item #
Sample Labels	Adhesive	
Thermometers	Jacketed, Digital, NIST traceable	
Sample storage cooling units	Capable of holding required storage temperatures	NA
COC forms	Chain of Custody Forms	N/A
SCUR forms	Sample Condition Upon Receipt	N/A
pH paper	Wide Range 0-14	CTL 921-10
Label Printer	Thermal Printer	NA
LIMS computer system	EPIC Pro	NA
Disposable pipettes	Glass	Baxter Scientific, P200-1
Sample containers	Glass or Plastic	C&G, QEC, Fisher
Temperature blank	Plastic	NA

10. Reagents and Standards

10.1. All reagents used in this procedure must be labeled with:

- 10.1.1. Laboratory reagent identification number;
- 10.1.2. Unless otherwise noted, the name and concentration of the reagent;
- 10.1.3. Date the reagent was received, opened and, as needed, prepared;
- 10.1.4. Person preparing reagent;
- 10.1.5. Expiration date.

10.2. Reagents: Table 10.1

Reagent	Formula	Concentration
Sulfuric Acid	H ₂ SO ₄	1:1
Nitric Acid	HNO ₃	1:1
Hydrochloric Acid	HCl	1:1
Sodium Hydroxide	NaOH	50% or Pellets
Sodium Thiosulfate	Na ₂ S ₂ O ₃ ·5H ₂ O	
Zinc Acetate Solution (for sulfide)		
Methanol	MeOH	Purge and Trap Grade
Ascorbic Acid (for cyanide)		
Sodium Bisulfate		
Ammonium sulfate/ ammonium hydroxide (for hexavalent chromium)		

10.3. For acids, bases and other reagents obtained from other laboratory departments, this information is located in the appropriate hardcopy or electronic standards/reagent preparation log. In the event that these reagents are managed within the Sample Receiving group, the department must maintain its own reagent preparation log.

10.4. Some Pace labs use preserved sample containers. In this case, documentation must be maintained for bottleware and preservation traceability.

11. Calibration and Standardization

11.1. Thermometers, IR-Guns, and other equipment used for measuring temperatures must be calibrated according to SOP S-GB-Q-030 **Support Equipment**, or its equivalent revision or replacement.

12. Procedure

12.1. Sample Receipt

12.1.1. The laboratory receives client samples via three major methods: mail/commercial delivery service, Pace Analytical courier/field services and hand delivery.

12.1.2. **Courier COC Procedures:** Pace labs use courier services that pick up client samples on either a regular schedule or on an as-needed basis as communicated by Project Managers (PMs) or by the client.

12.1.2.1. When the client is present during courier pick-up, the client signs the COC relinquishing custody to the courier. The courier signs the COC as accepting the samples and provides the client with a copy of the COC. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab.

12.1.2.2. If the client is not present during courier pick-up, the courier signs the COC as accepting the samples and leaves a copy of the COC for the client. If a client also has a sample log in use, the courier must sign and date the log when the samples are picked up. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab. The date/time of delivery to the lab by the courier is the official date/time received by the lab (analogous to the official date/time of receipt by an outside commercial carrier or courier).

12.1.2.3. To ensure the sample security, the Pace courier vehicle is locked at each client pick-up location. **IMPORTANT:** Pace Analytical courier/field services personnel must open the sample coolers and verify there is adequate ice in the coolers before transporting or shipping to the laboratory. An exception to this policy would be for coolers already custody-sealed by the client. These coolers are not to be opened except by the receiving lab personnel.

12.1.3. **Lab COC Procedures:** The COC (see example Attachment II) is signed immediately upon receipt of the samples from the client. If the client drops off the samples, a copy of the signed COC is given to the client at that time. If samples are received via commercial carrier or mail delivery, the COC should be signed immediately when the cooler or package is opened and ultimately placed in the project file. The delivery date and time is considered the date/time received.

12.1.3.1. **Samples Dropped Off:** Sample receiving personnel must review the COC for any evidence of rush turnaround requests, analyses with short hold times, or samples with very little hold time remaining. Projects that fall under these conditions must be given immediate attention. The PM responsible for that client must be alerted in the event that they have not already alerted the laboratory to the project as it may be possible that the client did not pre-schedule the project. Once the samples are received and logged into the LIMS, the sample technician and project manager will coordinate the notification and delivery of samples to the laboratory.

12.1.4. **Sample Acceptance Policy** - Copies of the Sample Acceptance Policy must be provided, in the form of a letter, fax, or e-mail to each client or sampler, as necessary. Samples are considered acceptable if they meet the criteria listed in the Sample Acceptance Policy (see Attachment I)

12.1.4.1. For WI drinking water samples: Samples that do not meet the criteria in the Sample Acceptance Policy will be rejected by sample custody. Sample custody will notify the PM and the client will be notified before proceeding with login. If the client wishes to proceed with analysis, the project manager will retain documentation of the request to proceed.

12.1.5. **Measuring temperature when temperature blank present:** Open the cooler and verify the temperature of the samples by taking the temperature of the temperature blank. The temperature of the cooler must be taken using a NIST-traceable thermometer. The thermometer is placed into the temperature blank. After 5 minutes, the thermometer is read to the nearest 0.5°C increment and recorded.

12.1.6. Measuring temperature when NO temperature blank present: If there is no temperature blank in the cooler, measure the temperature of a representative sample bottle or cooler melt water. A representative sample will reflect an “average” condition of the samples in the cooler and, depending on the manner in which they are packed, may not necessarily be in direct contact with the cooling material.

12.1.6.1. Procedure using a stick thermometer: If an IR gun is not used, the temperature of the cooler must be taken using a NIST-traceable thermometer. If there is no temperature blank, the thermometer is placed into the melt water of the cooler for approximately 5 minutes. After 5 minutes, the thermometer is read to the nearest 0.5°C increment and recorded. If no ice is present, a sample aliquot (non-volatile) is poured into a small container and the temperature of the sample is taken.

12.1.6.2. Procedure using an IR gun: If an IR gun is used, the temperature must be taken from an opaque surface such as the bottle label. Measurements taken through a transparent surface (clear or amber glass) may not be reliable and must incorporate a specific temperature correction factor for that surface reading.

12.1.7. Record the uncorrected and corrected cooler temperatures on the COC (example in Attachment II) and/or SCUR form (example in Attachment III) . In addition, record the type of “ice” used for packing the cooler (e.g., wet ice, “blue ice”, gel packs, etc.).

12.1.8. If samples within a project are spread over multiple coolers and one or more of the coolers are outside of the temperature criteria, then the contents of the cooler must be itemized and the samples and sample containers affected by the out-of-control temperature must be listed on the SCUR form for qualification in the final report. This itemization must be retained in the project file for future reference.

12.1.9. Unpack the cooler and COC. Organize the samples, grouped by client sample ID, according to the order on the COC. Review COC against samples to make sure the bottles received match the analysis requested. All anomalies must be recorded on the SCUR form.

12.1.9.1. If the lab receives coolers late or cannot unpack a cooler until the following day the following is performed:

12.1.9.1.1. The cooler is visually inspected and opened.

12.1.9.1.2. The COC is removed and signed.

12.1.9.1.3. The temperature of the cooler is taken and recorded on COC.

12.1.9.1.4. The COC is checked for short hold time samples. If short hold time samples are present, the cooler is processed immediately.

12.1.9.1.5. If short hold time samples are not present, the cooler and contents are placed into the designated Walk-in Cooler to be processed the following day.

12.1.10. For USDA samples, the cooler and all contents must be decontaminated (refer to Regulated Soil SOP for procedure). For non-USDA samples, discard any ice or water that remains in the cooler and the packing material used to secure the samples. Water or ice should be discarded down a drain that connects to the local sewer. Packing materials should be placed in the garbage. If a sample container was broken, the contents remaining in the cooler **MUST** be discarded in a manner consistent with the hazardous waste handling standard operating procedure.

12.1.11. pH Verification Instructions:

12.1.11.1. The pH of the sample must be verified on all preserved sample bottles requiring pH preservation (see exceptions below).

12.1.11.2. Open each preserved bottle (except as noted below). Use a new disposable pipette, a stirring rod or another inert utensil to withdraw a small portion of the sample. Dispense the aliquot on a sample specific pH strip and check the pH.

12.1.11.3. NOTE: Do not check the pH of samples for coliform, volatiles, Total Organic Carbon (TOC), Wisconsin Diesel Range Organics (WI-DRO), oil and grease, or hexane extractable materials (HEM). These analyses will be checked by the analyst at the bench and must not be opened by sample management personnel.

Table 12.1 – General pH Preservation Requirements by Preservative

Sample Preservatives	Sample pH Requirement
Hydrochloric Acid (HCl)	must be less than 2
Nitric Acid (HNO ₃)	must be less than 2
Sulfuric Acid (H ₂ SO ₄)	must be less than 2
Sodium Hydroxide (NaOH)	must be greater than 12
Zinc Acetate and Sodium Hydroxide (NaOH)	must be greater than 9

12.1.11.4. If the pH for a sample container that is supposed to be preserved is not within the required range, indicate the anomaly on the SCUR form or on the COC. If a sample does not require preservation, write N/A in the applicable section of the SCUR form.

12.1.12. pH Preservation Adjustments:

12.1.12.1. If a sample container does not meet the pH preservation required, the pH of the sample must be recorded on the COC or SCUR. Additional preservative is added so that the preservative content is < 1% of the sample container volume. For example:

12.1.12.1.1. For a 100mL container, a maximum of 1mL of preservative may be added;

12.1.12.1.2. For a 250mL container, a maximum of 2.5mL of preservative may be added;

12.1.12.1.3. For a 500mL container, a maximum of 5mL of preservative may be added;

12.1.12.1.4. For a 1L container, a maximum of 10mL of preservative may be added.

12.1.12.2. The appropriate preservative is added to the sample container, the sample is mixed and the pH is taken again. The new pH reading is also recorded on the COC or SCUR along with the amount, type and lot number of the preservative added. In addition, the sample container is marked with the preservative added, volume added, date, time and initials of the technician. For Metals analyses specifically, the lab must wait 24 hours after pH adjustment to pH < 2 before sample preparation can begin.

12.1.12.3. If unpreserved sample is received by lab and requires preservation, a clean container is used with appropriate preservation. An aliquant of unpreserved sample is poured out into preserved containers. Sample container is marked with a yellow sticker to notify lab that preservation was done in-house.

12.1.13. **Total Residual Chlorine Verification Instructions** - Total residual chlorine must be verified at the bench as required by the method or individual state regulatory agency for certain analyses (see Table 12.2). Do not check the sample bottles for those analyses listed in 12.1.11.3.

12.1.14.1. Open the appropriate sample container. Utilizing a new disposable pipette, stirring bar or other inert utensil; withdraw a small portion of the sample. Dispense the aliquot on a sample specific residual chlorine test strip.

12.1.14.2. If any chlorine is detected, regardless of amount, note the information on the COC, SCUR or analytical bench sheet.

Table 12.2 – Analyses requiring Residual Chlorine Verification

Analyses
Ammonia (NH ₃) EPA 350.1
Nitrate (NO ₃) EPA 353.2
Biochemical Oxygen Demand (BOD) SM5210B
Cyanides SM4500 CN and EPA 335.4
Dioxin 1613B
PBDE 1614
PCBs 1668A
EPA 508.1
EPA 549.2
EPA 515.3
EPA 548.1
EPA 608
EPA 610
EPA 625

12.1.14. **Checking for Sulfide in Cyanide analyses:** Test for sulfide by placing a drop of sample onto a piece of lead acetate paper. Darkening of the paper indicates the presence of sulfide. Follow specific method instructions for removing sulfide from samples.

12.1.15. Note any discrepancies pertaining to samples as defined by the sample acceptance policy detailed above on the COC or SCUR. Any discrepancies involving temperature, preservation, hold time, collection dates and times, sample volume, sample containers, and unclear analysis, must be reported to project management as soon as possible.

12.1.16. For short hold samples, the laboratory is notified and the samples are staged per section 12.2.

Table 12.3 – Analyses with Hold Times Less Than 72 Hours

Short Hold Time	Analyses	Details
15 minutes	Field Parameters	pH, Dissolved Oxygen, Residual Chlorine
8 Hours	Total/Fecal Coliform (MPN, MF), Enterococci, Fecal Streptococci MPN	Non-potable water only
8 Hours	Heterotrophic Plate Count (HPC)	
24 Hours	Hexavalent Chromium	
24 Hours	Fecal Sludge MPN	
24 Hours	Odor	
30 Hours	Total Coliform (Presence / Absence)	
48 Hours	Color	
48 Hours	MBAS	
48 Hours	Nitrate (unpreserved)	If Preserved, reported as NO3+NO2
48 Hours	Nitrite (unpreserved)	If Preserved, reported as NO3+NO2
48 Hours	Ortho -phosphate	
48 Hours	Settable Solids	
48 Hours	Turbidity	
48 Hours	VOA - Soils by Unpreserved EPA5035	Jars, Encores, Sleeves
48 Hours	Gross Alpha (NJ 48hr method)-waters	EPA NJAC 7:18-6
48 Hours	UV254	
48 Hours	Asbestos	
48 Hours	Chlorophyll A	48 hours to filtration
72 Hours	3030C Metals	
72 Hours	Volatiles – Air TO-18	Tedlar bag or equivalent

12.2. Sample Login

12.2.1. All samples received by the laboratory must be logged into the LIMS. Rush projects and/or projects with short holds should be prioritized. After these projects have been addressed, projects should be addressed on a first in, first out basis.

12.2.1.1. Samples must be logged into the LIMS so the samples can be uniquely identified (lab sample identification numbers). These lab sample ID numbers are used to track the prep and analysis activities of the samples, as well as identify the sub-samples, digestates, extracts, and other sample byproducts. This laboratory code maintains an unequivocal link with the unique client field sample ID code assigned to each sample.

12.2.1.2. Clients and Project Profiles are created in EPIC Pro as per Training Document T-ALL-IT-002 *Epic Pro 02: Client Setup*. Projects are logged as per Section 1 of Training Document T-ALL-IT-005 *Epic Pro: Login* (most current revisions or replacements).

12.2.1.3. Tests are assigned as per Training Document T-ALL-IT-002 *Epic Pro 02: Client Setup*, and as per Section 1 of Training Document T-ALL-IT 005 *Epic Pro: Login*.

12.2.2. Generate sample labels.

12.2.2.1. Local SRF generation is as per Section 3 of Training Document T-ALL-IT-005 *Epic Pro: Login*.

12.2.2.2. Local sample label generation is as per Section 2 of Training Document T-ALL-IT-005 *Epic Pro: Login*.

12.2.3. Attach the sample labels to the appropriate sample bottles.

12.2.3.1. Sample Management must make sure all sample ID's match COC, Samples are staged on the counter in rows by Sample ID. All sample containers are recorded on COC.

12.2.3.2. Sample labels are printed in Sample Management. One label is printed per sample container.

12.2.3.3. The label is placed onto the side of the container

12.2.4. If any samples require analyses performed outside of the laboratory, prepare the samples for subcontracting according to the procedures listed in the SOP describing the subcontracting of analytical services, S-GB-C-009 *Subcontracting Samples*, or equivalent revision or replacement.

12.2.5. The Project Manager, Project Coordinator, or designated Client Services personnel must review and verify the following information by comparing the COC to SRF. Some of this information may not be provided by the client and those fields should be left blank:

12.2.7.1. Report Recipient;

12.2.7.2. Invoice Recipient;

12.2.7.3. Additional Report Recipient;

12.2.7.4. PO#;

12.2.7.5. Project Name;

12.2.7.6. Project Number;

12.2.7.7. Requested Due Date;

12.2.7.8. Sample ID;

12.2.7.9. Matrix;

12.2.7.10. Collection Date & Time;

12.2.7.11. Received Date & Time;

12.2.7.12. Analysis: Double check compound lists;

12.2.7.13. Price;

12.2.7.14. Region Codes;

12.2.7.15. Work Region % Split (for Pace internal subcontracted work).

12.3. Sample Storage

12.3.1. Once unpacked, samples will be logged into the LIMS in a timely manner and returned to appropriate storage conditions as soon as possible. Labs must make every effort to keep samples under the required thermal conditions during the login process. For the exceptional case where samples are not logged in the day they were received, they must be stored under appropriate temperature-controlled conditions until login takes place. In all cases, the sample temperatures must be taken as soon after receipt as possible (before samples are placed into storage) and the samples stored so as to maintain the required storage conditions while awaiting log-in.

12.3.2. Once logged into the LIMS and labeled, samples are placed in the appropriate storage areas. Specific temperature requirements are outlined in the analytical methods, but general guidelines are outlined below:

12.3.2.1. Short hold samples are placed in the short hold storage area or delivered directly to the laboratory.

12.3.2.2. Biological tissue samples are staged by receiving date or project number on shelves in a freezer for all types of analyses.

12.3.2.3. Summa canisters and Tedlar bags are stored on designated shelving at ambient temperature.

12.3.2.4. Volatiles- Aqueous samples are stored by receiving date or by project number in a segregated volatiles cooler. Associated trip blanks are stored with the samples.

12.3.2.5. Volatiles- Soil and other solid samples received preserved in methanol are stored by receiving date or by project number in a segregated volatile cooler. Associated trip blanks are stored with the samples.

12.3.2.6. Volatiles- Soil and other solid samples received preserved with a stir bar, or deionized water and a stir bar, are stored by receiving date or by project number in a segregated volatiles freezer. Associated trip blanks are stored with samples.

12.3.2.7. Volatiles- Soil and other solid samples received in 4oz containers or similar bottleware must be preserved within 48 hours. In order to preserve these samples, it is necessary to collect a 5g aliquot of the sample and transfer it to a 40mL vial. One of the following preservation options must be utilized:

12.3.2.7.1. The 5g aliquot is preserved with a stir bar, 5mL of deionized water and a stir bar, or 5mL of sodium bisulfate and a stir bar and stored in a freezer until analysis, or;

12.3.2.7.2. Within 48 hours of collection in the field, the 5g aliquot must be immediately extracted with 5mL of methanol and stored in a segregated volatiles cooler until analysis, or;

12.3.2.7.3. Within 48 hours of collection in the field, the 5g aliquot can be preserved with 10mL of deionized water and a stir bar, stored in a segregated volatile cooler and analyzed within 48 hours of collection.

12.3.2.8. Volatiles- Soil and other solid samples received in Encore samplers must be managed within 48 hours of collection by freezing the Encore or extruding it.

12.3.2.8.1. If extruding the sample into a 40mL vial containing a stir bar or a stir bar and 10mL of deionized water, then the sample is stored in the segregated volatile freezer until analysis.

12.3.2.8.2. If extruding the sample into methanol, then the sample is extracted within 48 hours of collection and the sample is stored in a segregated volatile cooler until analysis.

12.3.2.8.3. NOTE: if samples are not received within 48 hours of collection or are not received with enough time to process the samples correctly within 48 hours of collection, this must be noted in a way that will be visible on the final report (e.g., footnote in LIMS).

12.3.2.9. General Chemistry/Semi-volatiles- Waters and other liquid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

12.3.2.10. General Chemistry/Semi-volatiles- Soils and other solid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

12.3.2.11. Metals Solids and Liquids: These samples are staged by receiving date or by project number on designated shelving in the laboratory or appropriate designated area. These samples may be stored at ambient temperature unless Mercury or Hexavalent Chromium analysis is needed. If Mercury or Hexavalent Chromium analysis will be performed, the samples are staged by receiving date or by project number in the appropriate sample storage cooler. Samples requiring low level mercury analysis by Method 1631 are taken to the clean room for preservation and ambient storage.

12.4. Sample Retention and Disposal

12.4.1. If samples must be returned to customers, the lab must take special care to ensure that the samples are not damaged during any handling, testing, storing, or transporting processes.

12.4.2. Samples may need to be retained longer than the normal sample retention time (45 days from sample receipt). Reasons for this extended sample retention include: customer, program, or contract requirements so that samples can be retained in a secure location for the customers that is designated as a long-term storage area.

12.4.3. Disposal of unconsumed samples: Refer to the laboratory SOPs regarding waste handling and disposal: *Waste Management and Handling* S-GB-W-001, and *Regulated Soil Handling* S-GB-S-001, or current revisions or replacements.

13. Quality Control

13.1. For any sample received at the laboratory that does not meet the sample acceptance, hold time or preservation criteria, the client must be contacted by project management and advised of the situation.

13.1.1. If the client instructs the laboratory to proceed with the analysis, all appropriate personnel/departments must be informed and the client approval must be documented on the SCUR or COC. Data will be appropriately qualified.

13.1.2. The client may also instruct the laboratory to preserve the samples at the laboratory prior to proceeding with analysis. This must be documented on the COC or the SCUR, and must be noted in the final laboratory report.

13.2. All supporting documentation related to sample custody must be retained by the laboratory. This includes: memorandums, fax transmissions, the original COC, all paperwork received with the COC, the completed SCUR form and copies of email transmissions. Please contact the laboratory QM/SQM for documentation retention time frames required.

13.3. Documenting discrepancies during receipt of samples:

13.3.1. The following are examples of client discrepancies that need to be documented on the appropriate paperwork (e.g., SCUR form):

13.3.1.1. Lost samples/insufficient sample volume;

13.3.1.2. Broken or missing bottles;

13.3.1.3. Missing COC;

13.3.1.4. Mislabeled bottles;

13.3.1.5. Preservation error;

13.3.1.6. Missing sample related details (date, time, sample type).

13.3.2. Pace sample management discrepancies will be documented on the SCUR form, COC or within the project files. Discrepancies attributable to errors and omissions on the part of the laboratory will be addressed and resolved through the formal corrective action process.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Hazards and Precautions - Use extreme caution in handling samples and wastes as they may be hazardous. Each reagent and chemical used in this method should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats, safety glasses, and ventilation hoods. SDS are on file and available to all personnel.

22.2. All personnel involved in sample management are responsible for complying with OSHA and DOT regulations. These regulations pertain to the safe handling and/or shipping of the chemicals specified in this procedure. Refer to the Sample Control Supervisor for any questions or concerns related to the safe handling and shipment of hazardous materials.

22.3. Other laboratory safety requirements are contained in the Chemical Hygiene Plan/Safety Manual. Immediate questions can also be addressed with the local Safety Officer.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

25.3. The NELAC Institute (TNI) Standard- most current version applicable to each lab.

25.4. SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA, current revision.

25.5. American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1995, Standard Methods for the Examination of Water and Wastewater, A.E. Greenberg, L.W. Clesceri, A.D. Eaton and M.A.H. Franson, eds., 19th ed., American Public Health Association, Washington D.C.

25.6. U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

25.7. U.S. Environmental Protection Agency, 1988, Methods for Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

25.8. Code of Federal Regulations- most recent version.

26. Tables, Diagrams, Flowcharts, and Validation Data

- 26.1. Attachment I: Sample Acceptance Policy, from form F-ALL-C-006.
26.2. Attachment II – Example Chain of Custody, F-ALL-Q-020.
26.3. Attachment III – Example Sample Condition Upon Receipt form, F-ALL-C-003.

27. Revisions

Document Number	Reason for Change	Date
SOT-ALL-C-001-rev.06	Cover page: revised the footer language and removed the uncontrolled document numbering line. General: made administrative edits that do not affect the policies or procedures within the document. Table 8.1: updated to match 2016 TNI Standard. Section 12.1.2.3: removed language regarding custody seals. Section 12.1.4: removed all Sample Acceptance Policy language in lieu of Attachment I. Sections 12.1.5, 12.1.6: reworded for clarity. Section 12.1.11, 12.1.13, 12.1.14: changed some text from red to black. Section 12.2.5: new section added requiring the state of origin to be documented. New Attachment I: Added Sample Acceptance Policy from form F-ALL-C-006. Old Attachment III: removed example SRF. Old Attachment IV: removed bottle/preservation table and all references to it within the SOP.	03Apr2017
S-GB-C-010-Rev.08	General: Added Pace-GB specific information which was in previous version of local SOP.	03Apr2017

Attachment I – Sample Acceptance Policy (from F-ALL-C-006)

In accordance with regulatory guidelines, Pace Analytical facilities comply with the following sample acceptance policy for all samples received.

If the samples do not meet the sample receipt acceptance criteria outlined below, the Pace facility is required to document all non-compliances, contact the client, and either reject the samples or fully document any decisions to proceed with analyses of samples that do not meet these criteria. Any results reported from samples not meeting these criteria are appropriately qualified on the final report.

Sample Acceptance Policy requirements:

1. Sample containers must have unique client identification designations, and dates and times of collection, that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC);
2. There must be clear documentation on the COC, or related documents such as the Sample Condition Upon Receipt (SCUR) form, that lists the unique sample identification, sampling site location (including state; some regulations may require city, county, etc.), date and time of sample collection, and name and signature of the sample collector;
3. There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, sample matrix, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.);
4. Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval;
5. Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval;
6. Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval;
7. Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval;
8. All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2°C of the required temperature, or within the method-specified range. For samples with a required temperature of 4°C, samples with a temperature ranging from just above freezing to 6°C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
9. For all compliance **drinking water** samples, analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).
10. Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.



Pace Analytical™
www.pacelabs.com


The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

[illegible]

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.08, 12-Oct-2007

Attachment III – Example Sample Condition Upon Receipt Form

Sample Condition Upon Receipt			
		Client Name: _____ Project # _____	
Courier: <input type="checkbox"/> Fed Ex <input type="checkbox"/> UPS <input type="checkbox"/> USPS <input type="checkbox"/> Client <input type="checkbox"/> Commercial <input type="checkbox"/> Pace Other _____		Optional: Proj. Due Date: _____ Proj. Name: _____	
Tracking #: _____			
Custody Seal on Cooler/Box Present: <input type="checkbox"/> yes <input type="checkbox"/> no		Seals Intact: <input type="checkbox"/> yes <input type="checkbox"/> no	
Packing Material: <input type="checkbox"/> Bubble Wrap <input type="checkbox"/> Bubble Bags <input type="checkbox"/> None <input type="checkbox"/> Other _____			
Thermometer Used _____		Type of Ice: Wet <input type="checkbox"/> Blue <input type="checkbox"/> None <input type="checkbox"/> Samples on ice, cooling process has begun	
Cooler Temperature _____ Temp should be above freezing to 6°C		Biological Tissue is Frozen: Yes <input type="checkbox"/> No <input type="checkbox"/>	
		Date and Initials of person examining contents: _____	
Chain of Custody Present: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		1.	
Chain of Custody Filled Out: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		2.	
Chain of Custody Relinquished: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		3.	
Sampler Name & Signature on COC: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		4.	
Samples Arrived within Hold Time: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		5.	
Short Hold Time Analysis (<72hr): <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		6.	
Rush Turn Around Time Requested: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		7.	
Sufficient Volume: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		8.	
Correct Containers Used: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		9.	
-Pace Containers Used: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A			
Containers Intact: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		10.	
Filtered volume received for Dissolved tests <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		11.	
Sample Labels match COC: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		12.	
-Includes date/time/ID/Analysis Matrix: _____			
All containers needing preservation have been checked. <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		13.	
All containers needing preservation are found to be in compliance with EPA recommendation. <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A			
exceptions: VOA, coliform, TOC, O&G, WI-DRO (water) <input type="checkbox"/> Yes <input type="checkbox"/> No		Initial when completed _____ Lot # of added preservative _____	
Samples checked for dechlorination: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		14.	
Headspace in VOA Vials (>6mm): <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		15.	
Trip Blank Present: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A		16.	
Trip Blank Custody Seals Present <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A			
Pace Trip Blank Lot # (if purchased): _____			
Client Notification/ Resolution: _____ Field Data Required? Y / N			
Person Contacted: _____ Date/Time: _____			
Comments/ Resolution: _____			

Project Manager Review: _____ Date: _____			

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

F-ALLC003rev.3, 11September2006



Pace Analytical Services, LLC Green Bay, WI
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469-2436
Fax: 920 469-8827


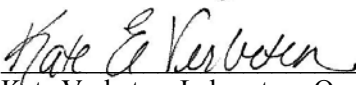
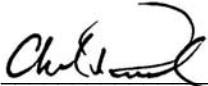
STANDARD OPERATING PROCEDURE

Total Cyanide using Micro-Distillation and SmartChem

Reference Method: SW846 9012B and EPA 335.4

SOP NUMBER:	S-GB-I-064-REV.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-064-Rev.03

APPROVAL

 _____ Nils Melberg, Laboratory General Manager	_____ Date
 _____ Kate Verbeten, Laboratory Quality Manager	_____ Date
 _____ Chad Rusch, Department Manager	_____ Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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1. Purpose/Identification of the Method

This Standard Operating Procedure (SOP) describes the analyses of Total Cyanide using Micro-Distillation and Analyzed by SmartChem Discrete analyzer using methods SW846 9012B and EPA Method 335.4.

2. Summary of Method

2.1 By means of a passive miniature distillation device, MICRO DIST, cyanide in the sample is released by digesting and acidifying cyanide complexes, and converting them to hydrocyanic acid (HCN). The cyanide ion is trapped in a 1.0 M sodium hydroxide absorbing solution, which is diluted to 0.25M solution during distillation. By means of discrete analysis, the 0.25M Noah distillate is converted to cyanogens chloride by reaction with chloramines-T, pyridine and barbituric acid to give a red-colored complex. The absorbance of this complex is measured at 570 nm by measuring the peak area resulting from the sample. The peak area is proportional to the concentration of the cyanide in the sample.

2.2 All samples must be distilled before analysis.

3. Scope and Application

3.1 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical and distillation methods.

3.2 Parameters: This SOP is applicable to the analysis of Cyanide in ground, surface, and saline waters, domestic and industrial wastes, soils, and TCLP, SPLP, ASTM extracts.

3.3 This SOP utilizes the SmartChem Discrete analyzer.

4. Applicable Matrices

4.1 Ground, surface, and saline waters

4.2 Domestic and industrial wastes

4.3 Soils and solid matrices

4.4 TCLP, SPLP, ASTM extracts

5. Limits of Detection and Quantitation

5.1 Current LOD and LOQ can be found in the Laboratory Information Management System (LIMS) - EpicPro.

5.2 Level of Detection (LOD): The LOD is determined by the 40CFR Part 136B MDL study. Once the 40CFR Part 136B MDL is determined it may be elevated if deemed unrealistic as demonstrated using method blank evaluations.

5.3 Level of Quantitation (LOQ): The LOQ is calculated as the LOD times 10/3. A realistic LOQ is typically near the lowest non-zero calibration point and higher than typical blank measurements.

6. Interferences

- 6.1 Most non-volatile interferences are eliminated or minimized by the distillation procedure. Some of the known interferences are aldehyde, nitrate-nitrite, and oxidizing agents, such as chlorine, thiosulfate, and sulfide.
- 6.2 Oxidizing agents such as chlorine decompose most cyanide. Test a drop of the sample with potassium iodine-starch paper (KI-starch paper) at time of collection; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color changes; then add an additional 0.06 g ascorbic acid per liter of sample.
- 6.3 False positive results may be obtained by samples that contain Nitrate/or nitrite. Sulfamic acid is added to all samples prior to distillation to eliminate this interference.
- 6.4 Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. If a drop of sample on lead acetate paper indicated the presence of sulfide, treat the stabilized sample (pH >12) with bismuth nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled.

7. Sample Collection, Preservation, Shipment and Storage

- 7.1 The lab provides appropriate bottle ware, including preservative, for requested testing. Where applicable, the bottle ware is demonstrated to be free of target analytes. When bottle ware not originating from the lab is used, the data may be qualified with either one or both of the following data qualifiers:
 - 7.1.1 Sample field preservation does not meet EPA or method recommendations for this analysis.
 - 7.1.2 Sample container did not meet EPA or method requirements.

7.2 Table 1: Sample Collection, Preservation and Handling

Matrix	Prep Method	Container(s)	Preservation Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SW-846 9012B EPA 335.4	250 ml plastic containers	pH >12; NaOH 14 days	On ice ≤6°C	≤6°C
TCLP, SPLP, ASTM	SW-846 9012B EPA 335.4	glass or plastic containers (when solid) Leachate is filtered into a 250 ml plastic container.	pH >12; NaOH 14 days to extract, additional 14 days to analyze (Total 28 days from collection)	On ice ≤6°C	≤6°C
Solid	SW-846 9012B	glass or plastic containers	None 14 days	On ice ≤6°C	≤6°C

8. Definitions

- 8.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.
- 8.2 Reagent Base Line (RBL) – Absorbance created when the diluent and method reagents are added to the cuvette. The diluent is a matrix match of the sample matrix.
- 8.3 Water Base Line (WBL) – Absorbance created when reagent grade water is added to the cuvette. The WBL is used to check the quality of the cuvette, condition of the filters, and lamp. If the value is too high or too low the cuvette will be rejected and will not be used in the analysis.

9. Equipment and Supplies

Table 2: EQUIPMENT

Equipment	Manufacturer	Model(s)/Catalog number •
SmartChem	Westco	200
Micro-Distillation	Lachat	Micro Distillation
Top loading Analytical Balance	Mettler	BA310P
1000 µL Pipette	Eppendorf	05-402-76 (Fisher Cat#)
500 µL Pipette	Eppendorf	05-402-72 (Fisher Cat#)
250 µL Pipette	Eppendorf	05-402-69 (Fisher Cat#)
Repipetors (0.5 - 5.0mL)	Barnstead/Thermolyne	13-687-33 (Fisher Cat#)
Vented Hood	Hamilton	
Spoonula (Lab Spoon)	FisherBrand or equivalent	14-375-10 (Fisher Cat#)
Gloves – Heat Resistant		
Cyanide Manifold	Lachat	10204-00-1-A
Interference filter – 570 nm	Lachat	

- Or equivalent

Table 3: Supplies

Supplies	Manufacturer	Catalog number •
Distillation tubes	Environmental Express	C8100C
Sample Cups 4mL	Unity	02-544-4
Volumetric Flasks	Fisher	Various
Parafilm	Fisher	
Teflon Chip	Fisher	
• Or equivalent		

10. Reagents and Standards

Table 4: REAGENTS

Reagent	Alias	Concentration	Directions found in:
Potassium Cyanide	Stock Standard	Neat	
Potassium Cyanide	Second Source Stock Standard	Neat	
Potassium Hydroxide		Neat	
Sodium Hydroxide		Neat	
Noah – 0.25 N	Carrier Solution	0.25 N	Table 6
Deionized Water			
Sodium Phosphate, Monobasic Monohydrate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$)		Neat	
Sodium dihydrogenphosphate Buffer	Buffer Solution	1 M	Table 6
Chloramine-T hydrate		Neat	
Chloramine-T Reagent	Color Reagent		Table 6
Barbituric Acid		Neat	
Pyridine		Neat	
0.0192N Silver Nitrate	LC226302	0.0192N	LabChem purchased from Fisher Scientific
Hydrochloric Acid		12 M	
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		Table 6
p-dimethylaminobenzalrhodanine	Indicating solution		Table 6
Standard Silver Nitrate Solution		0.0192 N	
Magnesium Chloride Hexahydrate		Neat	
Sulfuric Acid		Concentrated	
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Table 6
Nitrate Bismuth		Neat	
Lead Acetate Paper		Neat	
Sulfamic Acid		Neat	
Teflon chips		Neat	

Table 5: STANDARDS

Standard	Concentration	Direction found in:	Alias
Potassium Cyanide	1000 mg/L	Table 7	Stock Standard
Potassium Cyanide Must be different Lot # than Stock	1000 mg/L	Table 7	Second Source Stock Standard
Level 1 Calibration Standard	0.40 mg/L	Table 7	Cal Std. 1
Level 2 Calibration Standard	0.20 mg/L	Table 7	Cal Std. 2
Level 3 Calibration Standard	0.10 mg/L	Table 7	Cal Std. 3
Level 4 Calibration Standard	0.05 mg/L	Table 7	Cal Std. 4
Level 5 Calibration Standard and RLVS Standard	0.02 mg/L	Table 7	Cal Std. 5
Level 6 Calibration Standard	0.00 mg/L	Table 7	Cal Std. 6
Initial Calibration Verification – 1	0.10 mg/L	Table 7	ICV – 1
Initial Calibration Verification – 2	0.05 mg/L	Table 7	ICV – 2
Continuing Calibration Verification	0.20 mg/L	Table 7	CCV
Working Standard (1)	10 mg/L	Table 7	Working Standard (1)
Secondary Working Standard (1)	100 mg/L	Table 7	Secondary Working Standard (1)
Secondary Working Standard (2)	1.2 mg/L	Table 7	Secondary Working Standard (2)
Matrix Spike/Matrix Spike Duplicate – Liquid	0.10 mg/L	Table 7	MS/MSD-Liquid
Matrix Spike/Matrix Spike Duplicate – Solid	0.10 mg/L	Table 7	MS/MSD-Solid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Liquid	0.10 mg/L	Table 7	LCS/LCSD-Liquid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Solid	0.10 mg/L	Table 7	LCS/LCSD-Solid

Table 6: PREPARATION OF REAGENTS

Standard	Alias	Final Conc.	Directions	Final Volume
NaOH – 0.25 N	Carrier Solution	0.25 N	Weigh 10 g of neat NaOH into ~ 500 ml DI water in 1 L plastic container. Dilute to volume. Prepare fresh daily.	1000 ml
Sodium dihydrogenphosphate Buffer	Buffer Solution	1.0 M	Weigh 69 g of neat $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ into ~ 500 ml DI water in 1 L volumetric flask. Add 2.0 mL of concentrated Probe Rinse Solution. Dilute to volume. Shelf life = 3 months.	500 ml
Chloramine-T Reagent	Color Reagent		Weigh 0.4 g of neat Chloramine-T hydrate into ~ 50 ml DI water in 100 ml volumetric flask. Dilute to volume. PREPARE DAILY!	100 ml
Pyridine-Barbituric Acid Reagent	Pyridine- Barbituric Acid Reagent		MAKE UNDER HOOD!!!!!! Weigh 7.5 g barbituric acid into a 500 mL beaker Add 100 ml DI. Add 37.5 ml pyridine, mix to dissolve Add 7.5 ml conc. HCL. Transfer to 500 mL volumetric flask and dilute to volume. Shelf life = 3 months. Store in amber glass jar in the dark.	500 ml
p- dimethylaminobenzalrhodanine	Indicator Solution		Weigh 20 mg of neat p-dimethylaminobenzalrhodanine into a 100 ml volumetric flask. Dilute into 100 ml acetone. Shelf life = daily/when used.	100 ml
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Weigh 110.8 g DI water in beaker. Add 32.2 g Magnesium Chloride Hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$). Slowly add 139 g concentrated H_2SO_4 . Transfer to automatic pipette container. Cool. Shelf life = 3 months.	200 g

Table 7: PREPARATION OF STANDARDS

Standard	Alias	Final Concentration	Directions	Final Volume
Potassium Cyanide	Stock Standard.	1000 mg/L	POISON!!!! Weigh 2.51 g of neat KCN and 2 g of neat KOH into ~900 ml DI H ₂ O in 1 L volumetric flask. Dilute to volume. Shelf life = 6 months. Standardize against AgNO ₃ . If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Potassium Cyanide	Second Source Stock Standard	1000 mg/L	POISON!!!! Weigh 2.51 g of neat second source KCN and 2 g of neat KOH into ~900 ml DI H ₂ O in 1 L volumetric flask. Dilute to volume. Shelf life = 6 months. Standardize against AgNO ₃ . If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Working Standard (1)	Working Standard (1)	100 mg/L	Pipette 10 ml stock standard into 100 ml volumetric flask. Dilute to volume with DI H ₂ O. Prepare daily.	100 ml
Working Standard (2)	Working Standard (2)	1.2 mg/L	Pipette 1.2 ml Working Standard (1) into 100 ml volumetric flask. Dilute to volume with DI H ₂ O. Prepare daily.	100 ml
Secondary Working Standard (1)	Secondary Working Standard (1)	10 mg/L	Pipette 1 mL Potassium Cyanide (second source stock standard) into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Calibration standard – All other calibration points are diluted off of this standard by the instrument.	Cal. Std.	0.40 mg/L	Pipette 2.0 mL of Working Standard (2) into 6 ml distillation tube. Dilute to volume with DI H ₂ O. Prepare daily and distill.	6 ml
CRDL Standard	CRDL	0.020 mg/L	Pipette 0.25 mls of calibration standard into 5 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	5 ml
Calibration Blank	Cal. 0	0.000 mg/L	Use 0.25N NaOH only.	100 ml
Continuing Calibration Verification	CCV – 1	0.20 mg/L	Pipette 1.0 ml of Working Standard (2) solution into 6 ml distillation tube. Dilute to volume with DI H ₂ O. Prepare daily and distill.	6 ml
Initial Calibration Verification –1	ICV – A	0.10 mg/L	Pipette 1.0 ml of Secondary Working Standard (2) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Initial Calibration Verification –2	ICV – B	0.050 mg/L	Pipette 0.50 mL of Secondary Working Standard (1) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Matrix Spike/Matrix Spike Duplicate – Liquid	MS/MSD - Liquid	0.10 mg/L	Pipette 0.50 mL of Working Standard (2) into distillation tubes. Dilute to volume with sample. Prepare daily.	6 ml
Matrix Spike/Matrix Spike Duplicate – Solid	MS/MSD- Solid	0.10 mg/L	Pipette 0.50 mL of Working Standard (2) into sample tubes. Add solid sample. Dilute to volume with DI H ₂ O. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Liquid	LCS/LCSD- Liquid	0.10 mg/L	Pipette 0.50 mL of Working Standard (2) into sample tubes. Dilute to volume with DI H ₂ O. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Solid	LCS/LCSD- Solid	0.10 mg/L	Pipette 0.50 mL of Working Standard (2) into sample tubes. Dilute to volume with DI H ₂ O. Prepare daily.	6 ml

11. Calibration and Standardization

- 11.1 **Daily Calibration** – The SmartChem must be calibrated each time it is set up and at least every 24 hours that samples are analyzed. Calibration requires analysis of a minimum of 5 standards plus a blank due to the linear regression calibration used. The working range is from the current MDL to the highest standard in the calibration curve.
- 11.1.1 **Calibration Verification** – Each calibration must be verified by analyzing an Initial Calibration Verification (ICVA/ICVB) and Initial Calibration Blank (ICB). A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) must also be analyzed after each set of 10 samples.
- 11.1.2 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the lowest calibration point. The CRDL is used to demonstrate analyte recovery at the low end of the calibration curve.
- 11.1.3 **Acceptance Criteria** – The correlation coefficient of the response for elements requiring multiple levels must be ≥ 0.995 . The results of the ICV/CCV checks must agree within $\pm 10\%$. The results of the ICB/CCBs must be $< \text{LOQ}$.
- 11.1.4 Follow steps in section 12 to run calibration curve.
- 11.2 **Dilutions** -The SmartChem can automatically prepare diluted standards and samples, as required, to bring high concentration standards and samples into the working range of the method. See the SmartChem Operation Manual Chapter 3 for instructions on how to configure the SmartChem Parameter Method File.
- 11.3 **Standardization** – Cn standards made from neat sources must be standardized prior to use.
- 11.3.1 Add 1ml of stock/secondary source to a 100ml volumetric flask and dilute with 0.25N NaOH.
- 11.3.2 Pour solution in to a beaker and titrate using 0.0192N AgNO_3 , the starting color of the solution is yellow and will turn to a peach color indicating the end point. 1.0 mL of $\text{AgNO}_3 = 1.0 \text{ mg/L}$ of Cyanide.
- 11.3.3 A blank NaOH is also titrated (< 1 drop is needed for the color change).

12. Procedure

NOTE: Document all sample volumes, standards and reagents used in the distillation on the prep log.

- 12.1 Distillation Procedure - This section of the SOP details the steps in distilling the samples before analysis. Samples must be checked for sulfides before distilling. If sulfides are present and the samples treated with bismuth nitrate, the entire calibration curve must be similarly treated and distilled. Refer to section 6.4 for procedure. In the following procedure, **D** and **M** refer to the marks on the collector tube. **D** means “distillation” end, and **M** means “measuring” end.
- 12.1.1 Set the controller to 120°C ; allow the heater to warm up. The operating temperature of the distillation block needs to be verified annually.

- 12.1.2 Label tubes with the **M** end up, place as many properly labeled collector tubes as you have samples into the collector tube rack.
- 12.1.3 For liquid samples, using a transfer pipette place a drop of sample on lead acetate paper. If the paper darkens this indicates the presence of sulfide. Treat the stabilized sample (pH >12) with bismuth Nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled. Place 6 ml of sample into each properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

NOTE: If samples contain and are treated for sulfide, the entire calibration curve must also be similarly treated and distilled along with the samples.

- 12.1.4 For soil samples, weigh out ~0.20 g of soil or sludge to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water with a calibrated automatic pipette into each properly labeled sample tube. Add ~ 0.1 g sulfamic acid.
- 12.1.5 For liquid samples, prepare one method blank by adding 6 mls of DI water to properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.
- 12.1.6 For soil samples, prepare one method blank by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water with a calibrated automatic pipette into a properly labeled sample tube. Add ~ 0.1 g sulfamic acid.
- 12.1.7 For liquid samples, prepare one LCS (and one LCSD if needed/requested) by pipetting 6.0 ml of deionized water, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.
- 12.1.8 For soil samples, prepare one LCS (and one LCSD if needed/requested) by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.
- 12.1.9 For liquid samples, select one parent sample and prepare one MS/MSD pair per 10 samples by adding 6.0 ml of sample, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.
- 12.1.10 For soil samples, select one parent sample and prepare one MS/MSD pair by weighing out 0.20 g - 0.50 g of sample to the nearest 100th on a calibrated balance. The sample weight is dependent upon the sample matrix. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 mL of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.
- 12.1.11 Using a calibrated pipette, add 0.75 ml of 7.11 M sulfuric acid / 0.79 M magnesium chloride solution to the sample tube using the supplied automatic pipette.

- 12.1.12 Immediately push the **D** end of a Cyanide-1 collector tube over the open end of each sample tube to start the seal.
- 12.1.13 Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing, the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
- 12.1.14 The pressing motion should be a smooth constant pressure, which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
- 12.1.15 Put on heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
- 12.1.16 Set the timer for 30 minutes.
- 12.1.17 When 30 minutes is up, remove the first tube, use heat-resistant gloves, from the block and immediately pull its sample tube using a downward twisting motion as opposed to a sideways motion. You must remove the sample tube within 4 seconds of removing it for the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution into the 5 gallon waste container.
- 12.1.18 Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
- 12.1.19 Hold collector tube horizontally and rinse its walls with the distillate in order to homogenize it. Roll tube in order to collect all droplets from the walls of the tube. Return the tube to an upright position so the **D** end is up.
- 12.1.20 With the **D** end up, break the collector tube in half by pulling the **D** end hard toward yourself to break it, the twisting and tearing off the **D** end. Discard the **D** end.
- 12.1.21 In the **M** end of the tube dilute to the 6.0 ml mark with DI water. This results in the original sample volume, but now in 0.25 M NaOH. Pour in culture tube.
- 12.1.22 In the event that a distillation needs filtering prior to analysis, the batch QC must also go through the same filtration process. Document in the prep-log.
- 12.2 Basic System Operation (Analytical) - This portion of the SOP is designed to allow the user to set up and run a method. For a more detailed explanation of the many other options, the user should refer to the SmartChem Software Reference Manual. It is assumed that a method program has already been created for the chemistries you wish to run.
- 12.3 If not already on, turn on computer, printer, monitor, and the SmartChem instrument. The SmartChem on/off toggle switch is located on the left side of the instrument.
- 12.4 Log into the PC and Network. Click twice on SmartChem New and enter the username: *SmartChem New WESTCO* and password: *JOE*.

12.5 Build Sequence in LIMSLink of batched samples:

- 12.5.1 Go into LIMSLink and click on the “*Running Man*”.
- 12.5.2 Select the *Prep* method and click *Start New* followed by *OK*.
- 12.5.3 In the first two lines enter ICVA and ICVB followed ICB in the third line and CRDL in the fourth.
- 12.5.4 Click on Get Samples and enter the Batch, the Instrument ID (40WTA9) and click *OK*.
- 12.5.5 Repeat the last step for each batch of samples in the sequence.

12.6 SmartChem Sequence Loading and Analysis:

- 12.6.1 SmartChem Start-up procedure.
- 12.6.2 Go to Sample Entry
- 12.6.3 Double click on the desired Method to be run. For cyanide the method is CN-CN 335.4 Cyanide 20 to 400µg/L.
- 12.6.4 Click on Import from file button
- 12.6.5 Load Sequence file (create Run Plan) - open the SmartChem folder, scroll all the way to the right, and double click on *SmartChem*.
- 12.6.6 Save – Click on the red disk to save with the format *CN073112 40WTA9 WETA/EPA335.4 CCR*. 073112 is the month, day, year of the analysis. 40WTA9 is the instrument. WETA is the QUEUE and CCR is an example of analyst initials. Spaces must be used where they are. Click *OK*.
- 12.6.7 Save External File by clicking *YES* and Save the run plan as *CN073112*.
- 12.6.8 Go to System Monitor.
- 12.6.9 Select and Click on the Run Plan that was created.
- 12.6.10 Verify that the selected Run Plan is correct.
- 12.6.11 Load Samples, Standards, Controls, Diluent, and Empty Cups as displayed in the System Monitor.
- 12.6.12 Check the probe rinse, DI water, and Cleaning Solution bottles.

NOTE: When filling these solutions, pour slowly to minimize foaming. If filling is required while a run is in progress, do not lift siphon tube above the liquid surface. Insert a long stem funnel through the open port in the reservoir cap and fill the container slowly with the respective solution to minimize foaming.

- 12.6.13 Click Start in System Monitor to display user selectable options before beginning the analysis.

12.6.14 If a Wash Cuvette and/or WBL is required before the start of the analysis, then click on the appropriate button. It is recommended that a Wash Cuvette and WBL be run at the start of each day.

NOTE: It is recommended that a new WBL be run after a Wash Cuvette operation is performed and/or after the removal and/or replacement of the same or a new cuvette into the reaction tray.

12.6.15 Click the Start Button to begin analysis. A box will open. Make sure the Calibration and RBL options are selected for a run that is using a new calibration and click OK.

NOTE: Any daily or periodic maintenance must be recorded in the instrument daily logbook.

12.7 SHUTDOWN:

12.7.1 Click on EXIT icon

12.7.2 Turn off computer, printer and switch on surge suppresser.

13. Quality Control

13.1 Refer to the most current version of SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 Initial Calibration Verification (ICV)

13.2.1 The ICVA/ICVB must be analyzed before samples.

13.2.2 The ICVA/ICVB are not distilled.

13.2.3 Concentration must be within $\pm 10\%$ of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.4 If the ICVA/ICVB is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.2.5 The lot number of the Potassium Cyanide Standard used to make the ICVA/ICVB must be different from that of the calibration curve standards.

13.3 Continuing Calibration Verification (CCV)

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 The CCV is distilled with the batch.

13.3.3 Concentration must be within $\pm 10\%$ of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails, recalibrate and reanalyze all samples back to the last acceptable CCV.

13.3.4 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.3.5 The lot number of the Potassium Cyanide Standard used to make the CCV must be different from that of the ICV.

13.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)**

13.4.1 The ICB must be analyzed after the ICVA/ICVB and before samples. The CCB must be analyzed after the CCV and before samples.

13.4.2 The ICB and CCB are not distilled with the batch.

13.4.3 The absolute value must be < LOQ. When measurements are above the LOQ, terminate analysis, correct the problem, and the calibration re-verified. If the reset of the ICB/CCB fails, recalibrate and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.4.4 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

13.5 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Batch** – A batch will consist of up to 20 paid samples and will have at a minimum a method blank, laboratory control spike, and an MS/MSD pair at a frequency of 10% of the samples.

13.7 **Method Blank (MB)**

13.7.1 A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples. One MB must be distilled with batch.

13.7.2 The absolute value must be < LOQ.

13.7.3 Any samples digested with an unacceptable method blank must be re-prepped and analyzed unless the sample concentrations are less than the level being reported at or more than 10 times the value found in the method blank.

13.7.4 In those cases that LOD reporting is required; the MB must be evaluated to the LOD. For LOD reporting, an appropriate data qualifier is given to samples associated with \pm MB hits between the \pm LOD and \pm LOQ where the sample results are less than 10 times the value found in the method blank.

13.7.5 For negative instrument measurements >LOD and <LOQ qualify sample results that are non-detections and <10 times the measurement with "Analyte was measured in the associated method blank at a concentration of -#.# units." Make sure to enter the concentration and applicable sample units.

13.8 **Laboratory Control Sample (LCS)**

13.8.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one. The LCS must be distilled with batch.

- 13.8.2 Concentration must be within $\pm 10\%$ of the true value for waters. For soils, the concentration must be within calculated in-house limits or default limits of $\pm 20\%$.

$$\text{Accuracy Calculation: LCS \% Recovery} = \frac{\text{LCS}}{\text{SA}} * 100$$

Where; LCS= LCS Result
SA= Spike Added

- 13.8.3 When an LCSD is performed, the precision between the LCS and LCSD must be $\leq 20\%$ RPD.

The relative percent difference (RPD) is calculated as:
$$\text{RPD} = \frac{|\text{S} - \text{D}|}{(\text{S} + \text{D})/2} * 100$$

Where; S = LCS value, mg/L
D = LCSD value, mg/L

- 13.8.4 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.
- 13.8.5 If no errors are found and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.
- 13.8.6 If sufficient sample volume is not available, report the sample data with an appropriate (L) qualifier flag on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.9 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

13.9.1 One MS/MSD pair per up to 10 environmental samples or 10% frequency whichever is more frequent. The parent sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD. The source of the Ammonium Chloride Standard used to make the MS/MSD must be different from that of the calibration. The MS/MSD must be distilled with batch.

13.9.2 Both QC samples must be calculated for accuracy and precision.

Accuracy Calculation:
$$\text{Spike Percent Recovery} = \frac{(\text{SSR} - \text{SR})}{\text{SA}} * 100$$

Where; SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

The relative percent difference (RPD) is calculated as:
$$\text{RPD} = \frac{|S - D|}{(S + D)/2} * 100$$

Where; S = sample value, mg/L
D = duplicate value, mg/L

13.9.3 The concentration recovery must be within $\pm 10\%$ of the true value for waters. For soils, the concentration recovery must be within calculated in-house limits or default limits of $\pm 20\%$.

13.9.4 The precision between the MS and MSD must be $\leq 20\%$ RPD.

13.9.5 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results fall within the limits, accept the data and report without a qualifier flag.

13.9.6 If the four times the concentration of the spike is less than the level of the parent, accuracy need not be calculated.

13.9.7 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.10 Duplicate Sample (DUP)

13.10.1 Typically the method requirements for duplicate sample analysis are met with the MSD or LCSD, but based on client request a DUP may also be prepared and analyzed.

13.10.2 The DUP is evaluated for precision with the parent sample. If the RPD is outside 20% RPD criterion the parent sample and DUP are given an appropriate data qualifier. Parent sample is chosen at random or assigned by the client.

- 13.11 **Leach Blank** - The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date. The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, investigate and correct the problem, verify the calibration, and reanalyze all analytical samples analyzed.
- 13.12 **Hold Time** - When preparation of a sample exceeds 14 days past the time of collection, notify the project manager before proceeding. If a sample is run past 14 days after collection, flag the result with an appropriate data qualifier.
- 13.13 **Dilution** - If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with an appropriate data qualifier.
- 13.14 See attachments Table 8 and Table 9 for a summary of QC.

14. Data Analysis and Calculations

- 14.1 The instrument provides calculated sample results in µg/L. Calculations are only necessary if a dilution was used.
- 14.2 Liquid Calculation

$$\text{Raw Data Value (}\mu\text{g/L)} \times \text{Dilution Factor} / 1000 = \text{Cyanide (mg/L)}$$

- 14.3 Soil Calculation

$$\frac{\text{Raw Data Value (}\mu\text{g/L)} \times 0.006 \text{ L} \times \text{Dilution Factor}}{(0.20 \text{ \% solids in decimal form} \times 1000)} = \text{Cyanide (mg/Kg)}$$

15. Data Assessment and Acceptance Criteria for QC Measures

- 15.1 See Table 9: Data Assessment

16. Corrective Action for Out-of-Control Data

- 16.1 See Table 9: Data Assessment

17. Contingencies for Handling Out-of-Control or Unacceptable Data.

- 17.1 See Table 9: Data Assessment

18. Method Performance

- 18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.
- 18.2 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

- 18.3 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, Orientation and Training Procedures (current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager. A continuing demonstration of capability (CDOC) must be performed annually.
- 18.4 An annual Level of Detection (LOD) study will be completed per S-GB-Q-020, Determination of LOD and LOQ, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.
- 18.5 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, PE/PT Program (current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office. At a minimum, these are performed twice a year for the aqueous and soil matrices
- 18.6 A linear dynamic range study must be conducted at least once. The study is conducted for each element by analyzing increasing concentrations (at least 3 levels) until the results generated exceed $\pm 10\%$ difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that element. Once the linear dynamic range study determination is performed, keep the data, and then quarterly at a minimum verify with a single high point. Pace Analytical Services, LLC Green Bay, WI will not use any data over the highest calibration standard used. All samples will be diluted and reanalyzed that are over the calibration range.

19. Method Modifications

- 19.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 19.2 All major modification to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- 19.3 Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- 19.4 EPA Method 9012B is an aqueous method, the laboratory has modified the method to accommodate solid matrices.
- 19.5 EPA 335.4 sections 8.3 and 8.4 and EPA 9012B section 6.4 describe the thermal preservation as refrigeration at 4°C with no acceptable range. The lab practice is to have thermal preservation at <6°C. This is based on 40CFR Part 136, page 29808, footnote 18.
- 19.6 EPA Method 9012B and EPA 335.4 are described for macro glassware, the laboratory has chosen to use micro distillation equipment in place of the macro. All sample, reagent and standard volumes have been reduced accordingly
- 19.7 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.

20. Instrument/Equipment Maintenance

20.1 See Section 12.

21. Troubleshooting

21.1 See SmartChem Operating Manual.

22. Safety

22.1 Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, Waste Handling and Management (most current revision or replacement).

24. Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

24.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1 Pace Quality Assurance manual (most current revision or replacement).

25.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 SmartChem 200 Method 280-400E (Rev: April 2007)

25.4 *Micro Dist Method Cyanide-I*, Lachat Instruments, October 12, 2000

25.5 SW846 9012B, Revision 2.0, November 2004.

25.6 EPA 600 R-93-100, Revised August 1993, Method 335.4-1

25.7 Standard Methods for the Examination of Water and Wastewater, 18th Edition, p. 4-18,
 Methods 4500-CN⁻ A, B, C, and D (1992)

26 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

26.1 Table 8: **QC SUMMARY**

26.2 Table 9: **DATA ASSESSMENT/CORRECT ACTION**

26.3 Attachment I: Flowchart

27 Revisions

Revision Number	Reason for Change	Date
S-GB-I-064-Rev.02	Throughout Document: Updated to new format in SOP: S-GB-Q-017 <i>Preparation of SOPs</i> (most current revision or replacement). Updated SOP References throughout document. Table 7: Updated standard expiration dates. Section 11.1: Updated calibration curve fit to linear regression. Sections 11.2, 13.2 and 13.4: Added language for high/low ICVs (ICVA/ICVB). 12.1.10: Updated Soil weight language. Throughout document: Changed references to sea sand to Teflon chips, updated method references to EPA 9012B.	16Jan2014
S-GB-I-064-Rev.03	Section(s) 3.2, 4.1: Removed drinking water matrix. Section 5: Updated to LOD/LOQ language. Section 7.1: Updated bottleware information. Table 2, 3: Updated with current vendor information. Table 4: Added silver nitrate solution. Section 11.3: Added standardization of neat standards procedures. Section 13: Updated QC information. Section(s) 19.4, 19.5: Added.	17Nov2016
S-GB-I-064-Rev.04	Section 12.1.3: Stated to use a transfer pipette to test the pH	21Jun2017

Table 8: QC SUMMARY

Analytical Method ⇨ Quality Control Measure ⇩	EPA 335.4 SW846 9012B Frequency	Acceptance Criteria
Method Blank	<ul style="list-style-type: none"> One per batch of samples, up to 20 environmental samples, whichever is more frequent. Must be distilled with batch. 	<ul style="list-style-type: none"> Project Specific or Less than the LOQ
Laboratory Control Spike and Duplicate	<ul style="list-style-type: none"> One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent. An LCSD is required if MS/MSD is not performed or if requested by the client. Must be distilled with batch. 	<ul style="list-style-type: none"> Project Specific or For water 90 –110% with 20% RPD For solids 80 –120% with 20% RPD
Matrix Spike / Matrix Spike Duplicate	<ul style="list-style-type: none"> One pair per batch of samples, up to 10 environmental samples, whichever is more frequent. Must be distilled with batch. 	<ul style="list-style-type: none"> Project Specific or For water 90 –110% with 20% RPD For solids 80 –120% with 20% RPD
Initial Calibration	<ul style="list-style-type: none"> Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed. 	<ul style="list-style-type: none"> Correlation Coefficient of 0.995
CRDL	<ul style="list-style-type: none"> Analyzed after Initial Calibration, but before ICV 	<ul style="list-style-type: none"> 60-140%
Calibration Verification (ICV/CCV)	<ul style="list-style-type: none"> ICV – analyzed after calibration but before samples. The ICV is not distilled . CCV – analyzed after every 10 samples. The CCV is distilled with batch. 	<ul style="list-style-type: none"> Project specific or Recovery between 90 – 110%
Calibration Blank (ICB/CCB)	<ul style="list-style-type: none"> ICB – analyzed after ICV. The ICB is not distilled with batch. CCB – analyzed after every CCV. The CCB is not distilled with batch. 	<ul style="list-style-type: none"> Project specific or Less than RL
Leach Blank	<ul style="list-style-type: none"> The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date. 	<ul style="list-style-type: none"> Project specific or Less than LOQ

Table 9: DATA ASSESSMENT/CORRECTIVE ACTION

Data Assessment Measure	If these conditions are not achieved
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
Initial / Continuing Calibration Verification	• 5
Initial / Continuing Calibration Blank	• 6
Holding Time Compliance	• 7
CRDL/RLVS	• 8

1. If not <LOQ, verify by second analysis. If second analysis confirms contamination for target analyte at or greater than the LOQ, re-digest sample batch and batch QC provided sufficient sample volume remains. If insufficient sample volume remains, consult with project manager and client on how to proceed. For MB detections greater than or equal to the LOD, but less than the LOQ; qualify applicable sample results. For negative measurements more negative than the LOD, applicable data is given the following data qualifier: "Analyte was measured in the associated method blank at a concentration of -#.# units."
 - * For positive MB failures, samples that are non-detection need not be qualified. In addition, samples that are greater than 10 times the MB detection need not be qualified.
 - * For negative MB failures samples that are greater than 10 times the MB detection need not be qualified.
2. If the parent, MS, or MSD is greater than the reportable linear dynamic range, dilute and reanalyze the parent, MS, and MSD. If the concentration of the spike is less than 25% of the concentration of the parent the MS and MSD recoveries are not evaluated. Any failures resulting from this are qualified appropriately. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately qualify the parent sample if either the MS and/or MSD fail accuracy. If the MS and MSD fail precision control limits flag the parent with the appropriate precision data qualifier.
3. Verify failure by second analysis. If second analysis confirms LCS (LCSD) failure, re-digest sample batch and batch QC provided sufficient sample volume remains. If insufficient sample volume remains, consult with project manager and client on how to proceed.
4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.
5. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.
6. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.
7. Notify Project Manager by submitting a LabTrack Ticket and flag with the appropriate data qualifier.
8. If CRDL is outside the control limits reanalyze the CRDL to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. All applicable samples must be reanalyzed.

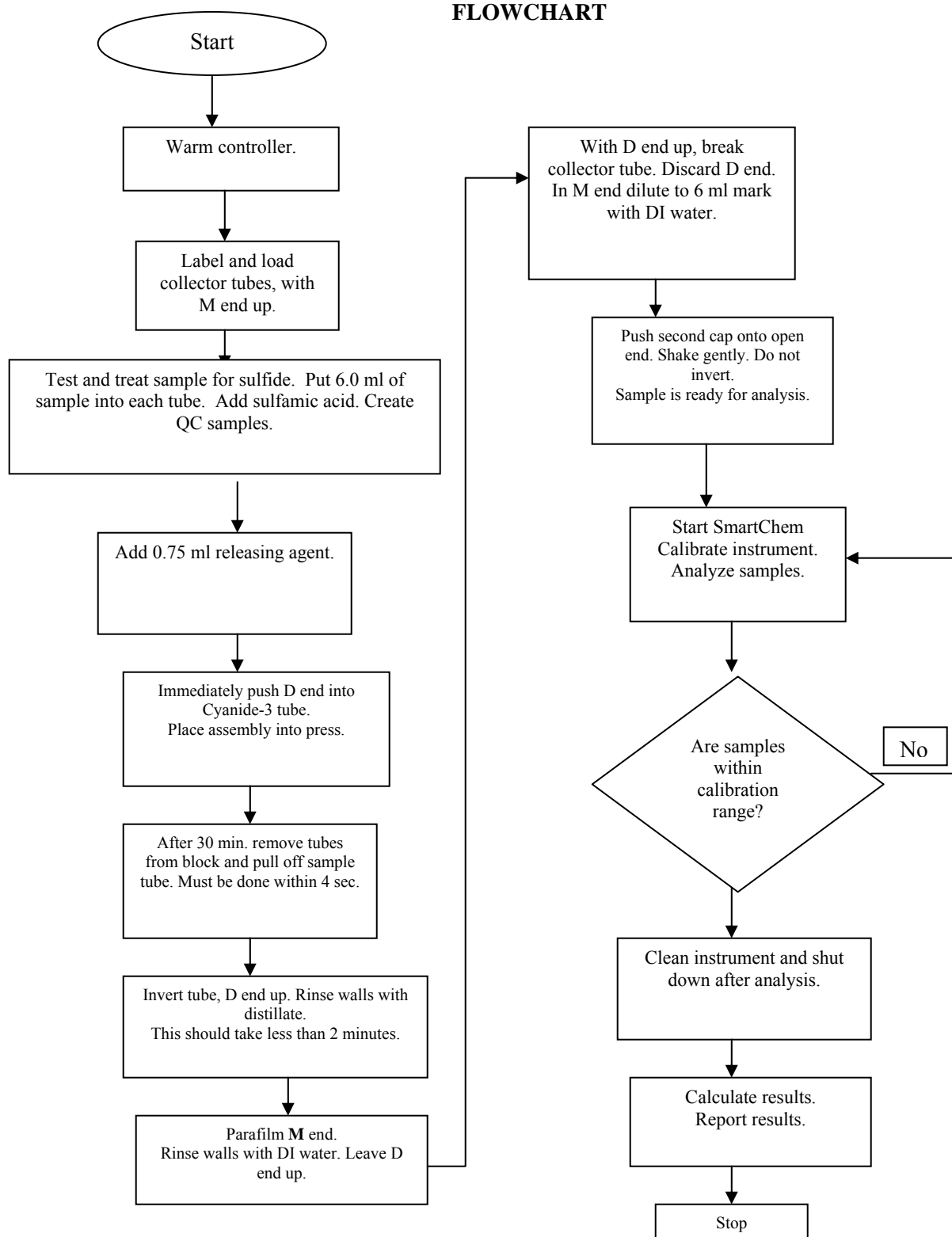
Table 10: Method Parameters

Type	End Point
Direction	Up
Decimals	2
Model	Linear
Filter 1	570 nm
Sample Blanking	No
Calibration Code	CYN4

Example of Extended Concentration Range 10 to 400 µg/L Without Sample Blanking					
Method Code: WCYN	Volume µL	Delay Time sec.	Read Time sec.	Rinse µL	Code
Range: 5-400 µg/L Cn Fluidics - Yes					
Sample	150				
Reagent 1 – Sodium Phosphate	63	36	0	0	CNSP
Reagent 2- Chloramine T	15	108	0	0	CNCL
Reagent 3 – Color Reagent	150	0	504	0	CNPY
Maximum Total Reaction Volume	670				
Minimum Sample and Reagent Volume	3				

Attachment I: Flowchart

FLOWCHART






STANDARD OPERATING PROCEDURE

TCLP - Toxicity Characteristic Leaching Procedure EPA SW846 1311 **SPLP – Synthetic Precipitation Leaching Procedure EPA SW846 1312** **ASTM - ASTM D 3987-85**

Reference Methods: EPA SW846 1311, EPA SW846 1312, ASTM D 3987-85

SOP NUMBER:	S-GB-I-067-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-067-Rev.02

APPROVAL

 _____ Nils Melberg, Laboratory General Manager	_____ 06/19/15 Date
 _____ Kate Grams, Laboratory Quality Manager	_____ 6/19/15 Date
 _____ Chad Rusch, Department Manager	_____ 6/19/15 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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1. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a laboratory specific procedure for extracting organics and inorganic compounds from samples using and meeting the requirements specified in ASTM D 3987-85 and also EPA SW846 1311(TCLP) and EPA SW846 1312(SPLP). This SOP covers bottle extraction and zero headspace extraction (ZHE).

2. SUMMARY OF METHOD

2.1 An aliquot of waste sample is tumbled in a sealed container containing extraction fluid to leach out compounds of interest from the sample. The extract then becomes the sample to analyze by a determinative method (volatiles, semi-volatiles, metals, wet chemistry).

3. SCOPE AND APPLICATION

3.1 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the preparation and analysis of samples using the leach methods within this facility.

3.2 Parameters: This SOP applies to the methods listed in Section 1.

4. APPLICABLE MATRICES

4.1 The leach methods are designed to determine the mobility of inorganic and organic contaminants present in liquid, solid, and multi-phase wastes.

4.2 Liquid wastes (below 0.5% dry solid material), after filtration through a 0.6 to 0.8 micron glass filter, are defined as the TCLP or SPLP extract. ASTM liquid samples are tumbled at a 1:20 ratio with reagent water.

4.3 For TCLP and SPLP wastes containing greater than 0.5% solids, the liquid portion, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted (tumbled) with an amount of extraction fluid equal to 20 times the weight of the solid phase. All ASTM samples are tumbled at a 1:20 ratio with reagent water.

4.4 Bottle extractors are used for non-volatile analytes and a special ZHE extraction device is used to tumble samples for volatile analysis.

4.5 For multi-phase samples being leached by TCLP and SPLP, the initial liquid phase and the solid phase extracted water are combined and analyzed as one sample. If the wastes are not compatible, they are analyzed separately and results are mathematically combined to give a volume-weighted average concentration. ASTM samples are homogenized and tumbled without being separated.

5. LIMITS OF DETECTION AND QUANTITATION

5.1 Not applicable to this SOP.

6. INTERFERENCES

No significant interferences are listed for the preparation of the TCLP extracts. Potential interferences during analyses are discussed in the individual determinative method SOP's.

7. SAMPLE COLLECTION, PRESERVATION SHIPMENT AND STORAGE

- 7.1** Samples are stored separately from all standards, reagents and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.
- 7.2** Liquid samples are collected in 1 Liter amber bottles with Teflon lined lids. Four to six liters may be needed for analysis. Preservations are not added to samples. After collection, samples are cooled to 4°- 6° C or lower, immediately after collection.
- 7.3** Solid samples are collected in 4 ounce wide-mouth bottles. At least 200g may be needed to perform the procedure. After collection, samples are cooled to 4°- 6° C or lower, immediately after collection.
- 7.4** Leach extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH <2, unless precipitation occurs. Extract should be preserved for other analytes according to the guidance given in the individual analysis method. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere to prevent losses.

- 7.5** TCLP, SPLP, ASTM extraction must be performed within the following times:

Sample Maximum Holding Times – Days				
	From: Field Collection To: TCLP Extraction	From: Leach Extraction To: Preparative Extraction/digest	From: Preparative Extraction/digest To: Determinative Analysis	Total Elapsed Time
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals	180	NA	180	360
Volatiles	14	NA	14	28
Cyanide	14	NA	14	28
Total Phenol, Anions (Wet Chem)	28	NA	28	56

- 7.6** Preservatives shall not be added to samples before tumbling.
- 7.7** TCLP extracts for metallic analyte determinations must be acidified with nitric acid to a pH of < 2. This is done after an aliquot has been spiked for the MS and/or MSD.
- 7.8** Extracts must be preserved and stored for analytes according to the guidance given in the individual analysis methods.

8. DEFINITIONS

8.1 Refer to the most current version section 10 of the Pace Quality Manual.

9. EQUIPMENT AND SUPPLIES - See Tables A and B for summary

10. REAGENTS AND STANDARDS - See Tables C and D for summary

10.1 **TCLP - Extraction fluid:** The pH of these fluids should be checked and recorded before each use; if the pH is outside of listed specifications or if impurities are found, the fluid should be discarded and remade. Shelf life = 1 week.

10.1.1 **Extraction fluid #1:** In 20 L carboy, add 20 L of DI water. Slowly add 114 ml glacial HOAc. Slowly add 130 ml 10 N NaOH. Cap carboy and mix well. The pH of this fluid must be 4.93 ± 0.05 standard units. If the pH is not within 4.93 ± 0.05 standard units, the fluid must be discarded and remade.

10.1.2 **Extraction fluid #2:** In 12 L carboy, add 10 L DI water. Slowly add 67.4 ml glacial HOAc dilute to volume. The pH of this fluid must be 2.88 ± 0.05 standard units. If the pH is not within 2.88 ± 0.05 standard units, the fluid must be discarded and remade.

10.2 **SPLP - Extraction fluid:** The pH of these fluids should be checked and recorded before each use; if the pH is outside of listed specifications or if impurities are found, the fluid should be discarded and remade. Shelf life = 1 week.

10.2.1 **Extraction fluid #1:** In 20 L car buoy, add DI water. Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until pH is 4.2 ± 0.05 standard units. If the pH is not within 4.2 ± 0.05 standard units, the fluid must be discarded and remade. The fluid is used to determine the leach ability of soil from a site that is east of the Mississippi River, and the leach ability of wastes and wastewaters.

10.2.2 **Extraction fluid #2:** In 20 L car buoy, add DI water. Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until pH is 5.0 ± 0.05 standard units. If the pH is not within 5.0 ± 0.05 standard units, the fluid must be discarded and remade. The fluid is used to determine the leach ability of soil from a site that is west of the Mississippi River.

10.2.3 **Extraction fluid #3:** Reagent water. The pH of this fluid must be within ASTM Type II water of 5.5 SU to 7.5 SU. This fluid is reagent water and is used to determine cyanide and volatiles leach ability.

10.3 **ASTM – Extraction fluid:** DI reagent water

10.4 Analytical Standards are not applicable to this SOP.

11. CALIBRATION

- 11.1** pH Meter shall be calibrated and verified as per directions in the most current version of SOP S-GB-I-071 *Measurement of pH in Water, Soil, and Waste*.
- 11.2** The balance calibration shall be verified as per the most current version of SOP S-GB-Q-030 *Support Equipment*.

12. PROCEDURE

12.1 ASTM Sample Preparation

- 12.1.1 Percent moisture is determined on each sample by Sample Management staff as per SOP S-GB-C-008, *Measurement of Percent Moisture in Soils and Solids*.
- 12.1.2 For free-flowing particulate solid wastes, obtain a sample of the appropriate size required by thoroughly mixing. A minimum sample size of 70 grams is recommended for each extraction; a sample size of 100 grams is commonly used in this method.
- 12.1.3 For field-cored solid wastes or castings, cut a representative section weighing approximately 100 grams for each testing. Shape the sample so the leaching solution will cover the material.
- 12.1.4 For fluid solid samples, mix thoroughly in a manner that does not incorporate air to assure uniformity before withdrawing a 100 gram sample for testing.

12.2 ASTM Extraction Procedure:

- 12.2.1 Weigh out 100 grams of sample into a 2-liter extraction bottle. Record the weight to the nearest 0.1 gram on the data sheet. If weights other than 100 grams are used, note on the data sheet.
- 12.2.2 Add to the container a volume of reagent water (in mL) 20 times the weight in grams of the sample. Use this dilution ratio for each sample. (Example: 100 grams sample = 2000 mL water).
- 12.2.3 Record the sample description, particle size, sample weight used for extraction, and amount of reagent water used in the Extraction Bench sheet Logbook.
- 12.2.4 Rotate at 29 +/- 2 rpm in rotating leaching apparatus continuously for 18 +/- 0.25 hours at 18 to 27°C. Record the tumbler ID and the min/max temperatures in the TCLP logbook. If the temperature is outside of specifications during the tumbling process, the temperature needs to be brought within control, and the entire batch needs to be re-prepared. Record start and stop time in logbook
- 12.2.5 Open container and observe sample. Record any physical changes in the sample and leaching solution.
- 12.2.6 Let the sample settle for 5 minutes. Separate the bulk of the aqueous phase from the solid phase by decantation, or filtration through a coarse filter paper.

- 12.2.7 Vacuum filter the liquid through a 0.45-um filter into a 2-liter flask. Document the lot ID of the filter used.
- 12.2.8 Measure the pH (record as final pH) of the extract immediately and preserve the volume of extract needed for the testing program as per Section 11.0. Record pH to the nearest 0.01 – pH units.
- 12.2.9 Refrigerate the extracts as soon as possible as 0° to ≤6°C.

12.3 ASTM ZHE Blank Vessel Preparation if Volatile Analysis is Required.

- 12.3.1 When the blank vessel is completely assembled and the lot ID of the filter paper documented, the vessel needs to be pressurized to eliminate all headspace before transferring the buffer solution. The vessel is connected to nitrogen by using the quick-disconnect Swagelok fitting found at the bottom of the vessel. When the quick-disconnect Swagelok is fitted, a distinct "click" should be heard. The two-way valve on the vessel's top plate is opened, and the vent relief valve on the vessel's bottom plate is closed. Approximately 10-20 psi of pressure is applied to push the piston completely to the top of the vessel, causing the vessel to "jump" slightly. The pressure is released in the vessel by venting the relief valve on the vessel's bottom plate.
- 12.3.2 Fill the transfer vessel to its capacity with DI water. The vessel's top plate is put on with the two-way valve opened. All the pressure release valves should be closed at this point. The transfer line is then attached tightly to the transfer vessel, and connected to the nitrogen tank. Pressure is applied, being careful not to exceed 50 psi. (Pressure greater than 50 psi may cause the glass fiber filter to break).
- 12.3.3 Slowly open the two-way valve, allowing the DI water to flow through the transfer line, thus eliminating the air that is in the line. Close the valve when the solution has reached the end of the transfer line, and loosely connect it to the blank vessel. Open both valves (two-way and vent relief) on the blank vessel, and open the two-way valve on the transfer vessel. Tighten the transfer line's connection to the blank vessel once you see the solution begin to leak. This prevents the addition of headspace to the blank vessel.
- 12.3.4 When there is no more flow through the transfer line, close the two-way valve on the blank vessel. Release the pressure on the transfer vessel. Pressurize to 40 psi and invert the blank vessel three times. Open the two-way valve slowly to allow any headspace to escape. As soon as liquid begins to elute without bubbles, close the top valve and shut off the nitrogen.

12.4 ASTM Solid Wastes Sample Preparation if Volatile Analysis is Required.

- 12.4.1 The sample is weighed out to 25 ± 0.5 grams, and is added to an empty ZHE vessel. The weight of the sample, the lot ID of the filter paper, the vessel number, and the tumbler ID are recorded in the ZHE Extraction Bench sheet logbook. The top plate of the vessel is closed to an even seal, and the same procedure used for the blank in step 12.3 is followed.

- 12.4.2 Add the DI water to the ZHE sample vessel via the transfer vessel according to the procedure described in the ZHE Blank Vessel section. The sample vessel is now ready to be tumbled after being pressurized to 5-10 psi.

Note: Assemble empty (dummy vessels) to fill any vacant spots in the tumbler for odd sample batches. Remove the legs from the vessels, if necessary, before tumbling. If the tumbler makes any bumping or banging noises when turned on, stop the tumbling, and re-adjust the vessels.

Note: When preparing actual samples, always vent into a hood. At a pressure of 5-10 psi, all samples and the blank are placed on the tumbler and rotated for 18 hours \pm 0.25 hours at 29 ± 2 RPM and 18° to 27°C . Record initiated and completed times, pressures, and temperatures in the ZHE logbook.

12.5 ASTM Extraction, Filtration, and Collection

- 12.5.1 After the samples have tumbled for 18 ± 0.25 hours, stop the tumbler and enter the leaching completed time and the min/max temperatures in the ZHE extraction logbook. The legs are re-attached to the ZHE vessels if necessary.

Note: Check the pressure gauges on the ZHE extractors and record in the ZHE logbook. If the ending pressure is less than 5-10 psi, the integrity of the vessel may have been breeched and analytes may have been lost. Check for damaged gaskets or weak or worn fittings. If the ending pressure is less than 5-10 psi, re-prepare and re-tumble affected samples.

- 12.5.2 Attach a Luer fitted 50 mL syringe to the top of each vessel. Connect each vessel to the Swagelok fitting from the bottom to the nitrogen tank and apply 20-30 psi of pressure. Slowly open the two-way valve on the top plate of the vessel to release the extract into the barrel of the syringe. Close the two-way valve and remove the syringe. Slowly transfer the extract into a 40 mL glass vial preserved with HCl. Fill three vials per sample.

Note: Add pressure slowly when necessary. There should be no gurgling noises, indicating an influx of air, as volatiles may be lost and the sample would then be invalid. Also, if the filter in the vessel breaks open, the sample becomes invalid. If either of these occurs, the entire extraction for the sample must be repeated.

12.6 TCLP and SPLP Procedure

- 12.6.1 Before leaching for TCLP and SPLP, perform these evaluations on a representative waste sample of at least 100 grams. If there is not at least 100 grams, get client consent before proceeding. **These test samples won't be extracted**, but are used for the preliminary evaluation.

- **Determination of matrix** of the waste sample
- **Determination of percent solids** of the waste sample (Done in Multi-Phase Worksheet if obviously not 100% solids)
- Determination of whether the waste sample contains insignificant solids and therefore, is its own extract after filtration, this is only done on liquid or **multi-phase** samples ($< 0.5\%$ (insignificant solids) or $\geq 0.5\%$.)
- Determination of if there is a need for **particle size reduction**

Determination of which **extraction fluid** is to be used for the extraction of the waste

12.7 TCLP and SPLP Determination of the matrix of the waste

12.7.1 The matrix of the waste must be determined, recorded, and reported to the analysts receiving the TCLP extracts for matrix spiking purposes. A matrix spike shall be performed for each matrix type.

12.7.2 The matrices are defined as:

12.7.2.1 Aqueous – Matrix that contains less than 0.5% solids.

12.7.2.2 Soil – Matrix consisting of natural ground.

12.7.2.3 Sludge – Matrix consisting of bio solids from a wastewater treatment facility.

12.7.2.4 Oil/Solvent – Matrix consisting of petroleum compounds immiscible with water

12.7.2.5 Other – Matrices that are none of the above, this matrix can include plant material, waste recycling/shredder material, concrete, etc. A matrix spike must be performed on each material determined as “Other”.

12.7.3 The extraction analyst must record the matrix for each sample as defined above in the TCLP/SPLP Extraction Bench Sheet Logbook. A copy of this record must accompany the sample extract to the analysts for matrix spiking purposes.

12.8 TCLP and SPLP Percent Solids of Waste – Use Multi-Phase Worksheet if sample has free liquid

12.8.1 **If the waste sample obviously will not yield free liquid during pressure filtration, it is treated as 100% solids. See Particle Size determination in section 12.9.**

12.8.2 If waste is a liquid or a multi-phase sample, separate the phases by filtration as follows:

12.8.2.1 Record the weight of the glass fiber filter and the container that will receive the filtrate. Assemble the filter and filtering assembly. Record the lot ID of the filter used.

12.8.2.2 Weigh out representative sample of waste, 100-gram minimum and record weight to 0.1 grams. Quantitatively transfer entire waste sample to filter holder. Spread evenly over surface of filter. Sample should be allowed to reach room temperature, as a refrigerated sample would yield less liquid.

NOTE: If some waste material adheres to container and cannot be transferred into filter holder (>1 percent of sample weight), subtract this residue weight from the original sample weight.

12.8.2.3 Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After 2 minutes at a maximum of 50 psi, the filtration is stopped.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging.

12.8.2.4 The material on the filter is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes or paint wastes, contain material that appears to be liquid, but may not filter. The material within the filtration device is defined as a solid. Do not replace the original filter with a fresh one during this determination.

12.8.2.5 Weigh the filled, filtrate container. Determine the weight of the liquid phase by subtracting the original weight of the filtrate container from the total weight of the filled, filtrate container.

12.8.2.6 Determine the weight of the solid phase by subtracting weight of the liquid phase from the total weight of the original sample aliquot.

12.8.2.7 Determine the percent solids using the following formula:

$$\text{Percent solids} = \frac{\text{weight of solid phase}}{\text{total weight of sample}} \times 100$$

12.8.2.8 Evaluation of Percent Solids

12.8.2.8.1 If the percent solid is greater than or equal to 0.5% and <2%, then proceed to step 12.8.2.9 (determination of percent DRY solids).

12.8.2.8.2 If the percent solid is greater than or equal to 2%, then proceed to step 12.9 (particle size reduction determination).

12.8.2.8.3 If the percent solid is less than 0.5%, the liquid phase (filtrate) is the TCLP extract: no extraction is needed. Proceed to step 12.11 to filter the sample.

12.8.2.9 Determination of percent DRY solids.

12.8.2.9.1 Dry the filter and solid phase at $100^{\circ} \pm 20^{\circ}\text{C}$ until two successive weightings yield the same value within ± 1 percent. Record the final weight in logbook.

12.8.2.9.2 Calculate the percent dry solids as follows:

$$\text{Percent DRY solids} = \frac{(\text{Wt of dry waste + filter}) - \text{Wt of filter}}{\text{Initial Wt of waste}} \times 100$$

12.8.2.10 Evaluation of percent DRY solids

- 12.8.2.10.1 If the percent DRY solid is less than 0.5%, the liquid phase (filtrate) is the TCLP extract: no extraction is needed, proceed to step 12.11 to filter the sample.
- 12.8.2.10.2 If the percent DRY solids are greater than or equal to 0.5%, then proceed to 12.9 (particle size reduction determination).

12.9 TCLP and SPLP Particle Size Determination

- 12.9.1 Particle size reduction is required unless the solid portion of the sample is capable of passing through a minimum 9.5mm sieve. If it will not pass through, the entire sample must be ground, crushed or cut until it will pass through. Use a fresh representative waste sample for this determination. If unable to reduce the particle size for a sample, document the reasoning and a detailed description of the sample matrix.

12.10 TCLP and SPLP Determination of Appropriate Extraction Fluid

12.10.1 TCLP

- 12.10.1.1 Extraction Fluid #1 is always used for Volatiles analysis. Do not perform the extraction fluid determination if only ZHE extraction is to be performed. Use a fresh portion of the solid phase of the waste for this evaluation.
- 12.10.1.2 Weigh out a small sub sample of solid phase. If necessary, reduce the particle size to approximately 1 mm in diameter or less and transfer about 5.0 grams into a 150 ml beaker. Document the amount of sample used.
- 12.10.1.3 If unable to reduce particle size for a sample to approximately 1mm, document the reasoning and detailed description of the sample matrix.
- 12.10.1.4 Add 96.5 mL DI water to the beaker, cover with a watch glass and stir vigorously for 5 minutes using a magnetic stirrer.
- 12.10.1.5 Measure and record the pH of the sample. If the pH is <5.0 use extraction fluid #1.
- 12.10.1.6 If the pH is >5.0, add 3.5 mL 1N HCl, document the 1N HCL in the logbook, slurry briefly, cover with a watch glass, heat to 50°C, and hold at 50°C for 10 minutes.
- 12.10.1.7 Let the solution cool to room temperature.
- 12.10.1.8 Measure and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

12.10.2 SPLP

- 12.10.2.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site west of the Mississippi River, extraction fluid #2 should be used.
- 12.10.2.2 For wastes and wastewater, extraction fluid #1 should be used.
- 12.10.2.3 For cyanide-containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

12.11 TCLP and SPLP Extraction Procedure

- 12.11.1 If the waste will obviously yield no liquid during filtration, weigh out a representative sub-sample, 100-gram minimum, record weight to nearest 0.1 gram.
- 12.11.2 If the waste is a liquid or a multi-phase sample, separate the phases as follows:
 - 12.11.2.1 Pre-weigh the container that will receive the filtrate. Assemble the filtering apparatus.
 - 12.11.2.2 If metals analysis is to be performed, use acid-washed filters. These can be purchased pre-washed or washed with 1 N HNO₃. Do not use acid washed filters for wet chemistry or semi-volatiles analysis. Therefore, if you need to leach for metals and wet chemistry or semivolatiles, you'll need to prepare two separate aliquots.
 - 12.11.2.3 Weigh out a representative sub-sample, 100-gram minimum, and record the weight to nearest 0.1 gram.

NOTE: If the sample contains <0.5 percent dry solids, the liquid phase is filtered and considered the extract. Enough sample volume should be filtered so that all necessary analyses can be performed on the filtrate. Likewise, use enough sample with >0.5 percent solids to generate enough solids for a representative extraction.

- 12.11.2.4 To aid filtration, allow slurries to stand to permit the solids to settle. The liquid phase may be filtered before the solid phase. (Centrifugation may be used to separate wastes that settle slowly.)
- 12.11.2.5 Quantitatively transfer entire waste sample to filter holder. Spread evenly over surface of filter. Sample should be allowed to reach room temperature as a refrigerated sample would yield less liquid.

NOTE: If some waste material adheres to container and cannot be transferred into filter holder, (>1 percent of sample weight), subtract this residue weight from the original sample weight.

- 12.11.2.6 Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When air begins to move through filter or when the liquid flow ceases at 50 psi, stop the filtration.

12.11.2.7 The material in the filter holder is the solid phase of the waste, and the filtrate is the liquid phase. Make sure there is enough solid phase of the sample to perform the extraction. The liquid phase may be analyzed separately or stored at 4°C and added to the extract from the rotated sample.

12.11.2.8 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the sample weight.

12.11.3 TCLP and SPLP: Determine the amount of extraction fluid by using the calculation:

$$\text{Weight of extraction fluid} = \frac{(20) \times (\% \text{ solids}) \times (\text{weight of waste filtered})}{100}$$

12.11.4 Since the density of the buffer solution is very near 1 gram/ml, it is assumed that 500 grams of buffer solution is equivalent to 500 ml of buffer solution.

12.11.5 Check pH of extraction fluids. This is documented as the initial pH for the leach blank using that fluid.

12.11.6 Slowly add the amount of appropriate extraction fluid to the extraction bottle. Close the bottle tightly and secure in agitation device.

12.11.7 Record the room temperature min/max for the duration of the sample rotation. The temperature of the room where the extraction takes place shall be maintained at $23 \pm 2^\circ\text{C}$. The room temperature is monitored continuously during rotation using a min/max thermometer. The room temperature is acceptable if it is within the range of 21-25°C.

12.11.8 Rotate at 30 ± 2 rpm for 18 ± 2 hours. Record the tumbler ID, rotational rate, the start, and stop times in logbook.

NOTE: Pressure may build up within the extraction bottle as agitation continues (e.g. calcium carbonate-containing wastes may evolve gasses such as carbon dioxide). To relieve excess pressure, the bottle may be periodically opened and vented into a hood (e.g. after 15 minutes, 30 minutes, 1 hour, etc.).

12.11.9 Following the 18 ± 2 hours extraction, filter the material in the extraction bottle through a new glass fiber filter. The glass fiber filter may be replaced to aid in filtration. If metals are to be analyzed, the filters should be acid rinsed with 1N HNO_3 . Record the lot ID of the filters used.

Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When air begins to move through filter or when the liquid flow ceases at 50 psi, stop the filtration.

12.11.10 Record the pH of the extract to the nearest 0.01-pH units.

12.11.11 Prepare the Leach extract as follows:

12.11.11.1 The metals extract must be acidified with HNO_3 to pH <2 after spiking the MS and/or MSD. This process step will be performed by metals staff. Metals and Wet Chemistry prep staff digest one Matrix Spike per waste stream. All other aliquots must be stored at 4°C until analysis.

12.11.11.2 If the waste contains no initial liquid phase, the filtered liquid material from 12.11.9 is considered the TCLP extract.

12.11.11.3 For multi-phase samples:

12.11.11.3.1 If separate phases are compatible, combine the filtered fluid with the “free liquid” obtained in step 12.11.2.7. This combined liquid is defined as the TCLP extract.

12.11.11.3.2 If the initial phase (free liquid) is not compatible with the filtered liquid from step 12.11.2.7, the liquids may be analyzed separately and the results combined mathematically as follows:

$$\text{Final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

V_1 = Volume of the first phase, in L

C_1 = Concentration of the analyte in the first phase, mg/L.

V_2 = Volume of the second phase, in L.

C_2 = Concentration of the analyte in the second phase, in mg/L.

12.12 TCLP and SPLP ZHE Vessel Preparation

- 12.12.1 For **volatile analysis**, the ZHE is used (extracts from the ZHE cannot be used for non-volatile analysis). Because the ZHE typically has a maximum capacity of 500mL, and because the amount of extraction fluid must be 20 times the sample weight, the maximum weight of a sample that can be used is 25 grams. A smaller sample may be used but the extraction fluid must still be 20 times the weight of sample used.
- 12.12.2 Do not allow the waste sample, the liquid phase (if any), or the extract to be exposed for any more time than is necessary.
- 12.12.3 When the ZHE vessel is completely assembled and the lot of the filter paper has been documented, it needs to be pressurized to eliminate all headspace before transferring the buffer solution. The vessel is connected to nitrogen by using the quick-disconnect Swagelok fitting found at the bottom of the vessel. When the quick-disconnect Swagelok is fitted, a distinct "click" should be heard. The two-way valve on the vessel's top plate is opened, and the vent relief valve on the vessel's bottom plate is closed. Approximately 10-20 psi of pressure is applied to push the piston completely to the top of the vessel, causing the vessel to "jump" slightly. The pressure is released in the vessel by venting the relief valve on the vessel's bottom plate. Make sure to shut off the Nitrogen source before venting.
- 12.12.4 Fill the transfer vessel to its capacity with buffer solution. The vessel's top plate is put on with the two-way valve opened. All the pressure release valves should be closed at this point. The transfer line is then attached tightly to the transfer vessel, and connected to the nitrogen tank. Pressure is applied, being careful not to exceed 10 psi.
- 12.12.5 Slowly open the two-way valve, allowing the buffer solution (fluid #1) to flow through the transfer line, thus eliminating the air that is in the line. Close the valve when the solution has reached the end of the transfer line, and loosely connect it to the ZHE vessel. Open both valves (two-way and vent relief) on the ZHE vessel, and open the two-way valve on the transfer vessel. Tighten the transfer line's connection to the ZHE vessel once you see the solution begin to leak. This prevents the addition of headspace to the ZHE vessel.
- 12.12.6 When there is no more flow through the transfer line, close the two-way valve on the ZHE vessel. Release the pressure on the transfer vessel. Pressurize to 10 psi and invert the ZHE vessel three times. Open the two-way valve slowly to allow any headspace to escape. As soon as liquid begins to elute without bubbles, close the top valve and shut off the nitrogen.

12.13 TCLP and SPLP Solid Wastes Sample Preparation (ZHE)

- 12.13.1 After the sample has been processed through steps 12.7 through 12.10, and there is no liquid in the sample, the sample is said to contain 100% solids, by definition. The 100% solids sample requires 500 grams of buffer solution. (Since the density of the buffer solution is very near 1 gram/mL, it is assumed that 500 grams of buffer solution is equivalent to 500 mL of buffer solution).

- 12.13.2 The sample is weighed out to 25 ± 0.5 grams, and is added to an empty ZHE vessel. The weight of the sample and the vessel number are recorded in the ZHE logbook. The top plate of the vessel is closed to an even seal, and the same procedure used for the blank in step 12.3 is followed.

12.14 TCLP and SPLP Liquid Wastes Sample Preparation (ZHE)

- 12.14.1 For liquid samples, weigh the glass fiber filter, document the filter lot ID, and record the weight in the ZHE Extraction Bench Sheet logbook before the ZHE vessel is assembled. Pour the sample into the vessel and close the top plate tightly. Attach a Luer fitting to the two-way valve on the top plate. Connect a small length of tygon tubing to the Luer fitting.
- 12.14.2 Pressurize the vessel to 10-20 psi and allow the sample to filter through. Collect the sample in a 40 mL HCL vial. Slowly increase the pressure, by 10-psi increments, up to 50 psi, and hold for 2 minutes. Allow the fluid to collect between pressure increases.
- 12.14.3 After filtration: Very carefully remove the filter so as not to tear it! Dry the filter in an oven at 80-120° C for 24 hours. When the weight does not vary by more than one percent over a one-hour period, record the last value obtained. Use it to determine the percent dry solids. If the percent dry solids is <0.5%, the fluid eluted is the ZHE extract and no buffer solution is required.

Note: A sample containing particulate matter that immediately clogs the filter during the filtration process, while obviously mostly liquid, is considered to be 100% solids.

12.15 TCLP and SPLP Multi-phase Liquids or Solids Saturated With Liquid

- 12.16.1 According to SW846 Method 5030 (7.3.3.2.1): "The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula."
- 12.16.2 Place 25 ± 0.5 grams of sample into a ZHE vessel. Pressurize the vessel 10-20 psi. Any liquid that elutes is collected into a tarred beaker. Slowly increase the pressure, by 10-psi increments, up to 50 psi. Allow the fluid to collect into the beaker between pressure increases. After two minutes at 50 psi, weigh the beaker and record the weight in the ZHE Extraction Bench Sheet logbook.
- 12.16.3 For multi-phasic wastes, the fluid collected in the beaker is weighed and subtracted from the total sample weight to determine the percent solids.

$$\frac{W_s - W_f}{W_s} \times 100 = \% \text{ Solids}$$

Where:

W_s = Weight of the sample

W_f = Weight of the eluted fluid

If no liquid elutes out of the vessel, the sample is considered to be 100% solids. The set-up can be used--as it is--for the ZHE extraction. Follow the steps in 12.13.

- 12.16.4 After collecting the fluid portion of the sample, set up the ZHE apparatus - a second time with a sample size computed according to the following formula

$$\frac{25}{\% \text{ Solids}} \times 100 = \text{Weight of Sample to be extracted (in grams)}$$

For example, if the sample weight equals 25.0 g, with 5.0 g of fluid eluted, the % solids equal 80%. The amount of sample to be extracted would be 31.2 g.

- 12.16.5 Collect the fluid via pressure filtration, as described in step 12.13, into a tarred container. Record the weight of the fluid in the ZHE Extraction Bench Sheet logbook. Store the fluid at $\leq 4^{\circ}\text{C}$ until analysis. Add the buffer solution to the vessel after the fluid has been eluted. Calculate the amount of buffer solution needed is according to the following formula:

$$\frac{20 \times \% \text{ Solids} \times \text{Weight of Sample}}{100} = \text{Weight of buffer solution}$$

For example, if 31.2 g of sample were added to the vessel, with a % solids value of 80%, the weight of buffer solution needed would be 499 g. Again, since the density of the buffer solution is very near 1 gram/mL, it is assumed that 499 grams of buffer solution is equivalent to 499 mL of buffer solution.)

- 12.16.6 Add the buffer solution (fluid #1) to the ZHE sample vessel via the transfer vessel according to the procedure described in the ZHE blank section. The sample vessel is now ready to be tumbled.

Note: Assemble empty (dummy vessels) to fill any vacant spots in the tumbler for odd sample batches. Remove the legs from the vessels, if necessary, before tumbling. If the tumbler makes any bumping or banging noises when turned on, stop the tumbling and re-adjust the vessels.

Note: When preparing actual samples, always vent into a hood. At a pressure of 10 psi, all samples and the blank are placed on the tumbler and rotated for 18 hours (± 2 hours) at 30 ± 2 RPM. Record initiated and completed times and temperatures in the ZHE Extraction Bench Sheet logbook.

12.17 TCLP and SPLP Extraction, Filtration, and Collection

- 12.17.1 After the samples have tumbled for 18 ± 2 hours, stop the tumbler and enter the leaching completed time, final pressure, and min/max temperatures in the ZHE Extraction Bench Sheet logbook. The legs are re-attached to the ZHE vessels if necessary.

Note: Check the pressure gauges on the ZHE extractors and record in the ZHE logbook. If the ending pressure is less than 10 psi, the integrity of the vessel may have been breeched and analytes may have been lost. Check for damaged gaskets or weak or worn fittings and redo the ZHE extraction

Attach a Luer fitted 50 mL syringe to the top of each vessel. Connect each vessel to the Swagelok fitting from the bottom to the nitrogen tank and apply 10 psi of pressure. Slowly open the two-way valve on the top plate of the vessel to release the extract into the barrel of the syringe. Close the two-way valve and remove the syringe. Slowly transfer the extract into a 40 mL glass vial preserved with HCl. Fill three vials per sample. Gradually apply pressure of 1-10 psi until sample extract passes through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

Note: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When the liquid flow ceases at 50 psi, stop the filtration.

Note: Add pressure slowly when necessary. There should be no gurgling noises, indicating an influx of air, as volatiles may be lost and the sample would then be invalid. Also, if the filter in the vessel breaks open, the sample becomes invalid. If either of these occurs, the entire extraction for the sample must be repeated.

12.17.2 For multi-phase samples:

The liquid obtained prior to extraction (from a multi-phase sample or solid sample saturated with liquid) must either:

- If miscible, combine with the extract and analyzed as one.
- If non-miscible, analyze separately from the extract, mathematically combining the results using the following volume-weighted average formula.

$$\frac{V_1 C_1 + V_2 C_2}{V_1 + V_2} = \text{combined result}$$

Where:

V_1 = the volume of the liquid phase.

V_2 = the volume of the ZHE extract.

C_1 = the concentration of the analyte in the liquid phase.

C_2 = the concentration of the analyte in the ZHE extract.

12.17.3 Store the extracts at 4°C and analyze according to the specific analytical methods used.

13 QUALITY CONTROL

13.1 A method blank comprised of each Extraction fluid used in the set is analyzed with each extracted batch not to exceed 20 samples.

13.2 Matrix spikes are not performed at the time of extraction but must be performed at the time of analysis. Spiking is performed as one spike per waste type (e.g., sludge, soil, aqueous, oil/solvent, etc.).

13.3 Check rotation of the tumbling apparatus per use and record in the logbook. Must be 30±2 RPM. A supervisor should be alerted if the rotation speed is unacceptable and corrective actions must be taken to make the speed acceptable.

13.4 Check start and stop room temperatures. The temperature of the room where the extraction takes place shall be maintained at $23 \pm 2^{\circ}\text{C}$. If outside this temperature range, the leach procedure must be redone.

13.5 The ZHE extractors should be checked each use for leaks by either pressuring to 50psi or then re-checking the pressure later to see if it stayed the same, or pressurize the ZHE and submerge it in water and look for escaping air bubbles.

13.6 Blanks should be rotated among all vessels. Record vessels used for blanks in the Bottle Blank Location logbook (see attachment).

14 DATA ANALYSIS AND CALCULATIONS

14.1 See Section 12 for applicable calculations.

15 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1 Not applicable to this SOP.

16 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Not applicable to this SOP.

17 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 Not applicable to this SOP.

18 METHOD PERFORMANCE

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager. Note: IDoC for TCLP extraction is documented by demonstration of the trainee to the trainer.

18.1.3 MDLs are not applicable.

18.1.4 Proficiency Test Studies (PT's) are not applicable.

19 METHOD MODIFICATIONS

19.1 Difficult matrices including, but not limited to: paper, lead paint chips, and tire is processed through the leach procedures.

20 INSTRUMENT/EQUIPMENT MAINTENANCE

20.1 Any daily or periodic maintenance must be recorded in the instrument daily logbook. Additional information may be found in the most current revision of SOP: S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance*.

21 TROUBLESHOOTING

21.1 Please see the instrument manual for information on instrument troubleshooting.

22 SAFETY

22.1 Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23 WASTE MANAGEMENT

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24 POLLUTION PREVENTION

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation

24.2 The laboratory Chemical Hygiene Plan and Safety Manual contains additional information on pollution prevention.

25 REFERENCES

“Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”, SW-846, Method 1311; 3rd edition; December 1996.

Annual Book of ASTM Standards, Volume 11.04, Method D 3987-85 (approved October 21, 1985, Published March 1986), Reapproved 2004.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, November 2000, Method 1312

26 TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

Table A – Summary of Equipment

Table B – Summary of Supplies

Table C – Summary of Reagents

Table D – Preparation of Reagents

Attachment I: Glass Bottle - Blank Rotation Log

Attachment II: Plastic Bottle - Blank Rotation Log

Attachment III: ZHE Vessel - Blank Rotation Log

Attachment IV: Flow Charts

27 Revisions

Document Number	Reason for Change	Date
GB-I-067-Rev. 00	First Issue Merged SOPs: S-GB-I-025 <i>Toxicity Characteristic Leaching Procedure (TCLP)</i> , S-GB-I-057, <i>SPLP – Synthetic Preparation Leaching Procedure</i> , and S-GB-I-058, <i>ASTM – Shake Extraction of Solid Waste with Water</i> .	05July2010
S-GB-I-067-Rev.01	Updated Signature Page Updated SOP references throughout document. Section 7.7 and 12.78: Added requirement to spike MS/MSD prior to preservation. Section 12.3.2.8 and 12.3.2.10: Added additional information to clarify the evaluation of DRY results and % solids. Section 12.7.4: Removed Tempscribe reference. Table A: Added Borosilicate Glass Extraction vessels for SVOA samples.	26Sept2012
S-GB-I-067-Rev.02	Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 <i>Preparation of SOPs</i> . Updated SOP per audit finding	13May2013
S-GB-I-067-Rev.03	Updated section references throughout document. Section 12.8: Removed reference to 100% Solids and updated to free liquids. Section 12.8.2.10.3: Removed. Section 12.9.1: Updated information on particle size reduction. Section 13: Updated TCLP Tumbler Blank Rotation to per-use from periodically. Section 22.1: Updated MSDS to SDS Section 23.1: Updated SOP reference Attachments: Added new blank rotation forms.	19Jun2015

Table A
EQUIPMENT

Equipment	Manufacturer	Model(s)	Notes:
Millipore Rotary Agitators	Millipore or equivalent	YT31 ORA HW	
3000mL Borosilicate Bottles	Environmental Express	BK3000-1	For SVOA samples
Rotary Agitator Bottles	Millipore or equivalent	YT30 09G BT	For Metal/Wet
ZHE Vessels			
Hazardous Waste Filtration Vessel	Millipore or equivalent	YT30 090 HW	
Funnel	-	-	
Beakers	VWR	13912-207	
pH Meter	Corning	PH 245	
Balance	Ohaus	Precision Standard	
Syringes	GasTight	1050	
Watch glass	-	-	
Forceps			
Magnetic Stirrer	Corning	PC 353	
Stir Bars	-	-	
Spoonula (Lab Spoon)	FisherBrand	14-375-10 (Fisher Cat#)	
100 mL Graduated Cylinder	-	-	
2000 mL Graduated Cylinder	-	-	
Carboys	-	20 liter and 12 liter	
Vacuum Pump	-	-	
Min/Mas Thermometer	-	-	
Thermometer	-	-	
Stirring Hotplate	Corning	Stirrer hotplate	
1L Volumetric Flask	-	-	
Heated Water Bath	-	-	

*- Or Equivalent

Table B
SUPPLIES

Supplies	Vendor	Catalog #
142 mm Acid Washed TCLP filters	Environmental Express	FG77142MM
90 mm TCLP filter	Environmental Express	FG77090MM
40 mL HCL Glass VOA vials	QEC	
Compressed Nitrogen Gas	AirGas*	

Or Equivalent


Table C
REAGENTS

Reagent	Alias	Concentration	Directions found in:
Reagent Water	ASTM Type II	5.5SU - 7.5SU	-
Sodium Hydroxide		10 N	Table D
Nitric Acid		1 N	Table D
Sulfuric Acid			
Glacial Acetic Acid	HOAc	Concentrated	
Hydrochloric Acid		1 N	Table D
TCLP Extraction Fluid #1	TCLP Fluid #1	pH 4.93 \pm 0.05	Table D
TCLP Extraction Fluid #2	TCLP Fluid #2	pH 2.88 \pm 0.05	Table D
Sulfuric Acid/Nitric Acid	NA	60/40 weight percent mixture	Table D
SPLP Extraction Fluid #1	SPLP Fluid #1	pH 4.2 \pm 0.05	Table D
SPLP Extraction Fluid #2	SPLP Fluid #2	pH 5.0 \pm 0.05	Table D

Table D
PREPARATION OF REAGENTS

Reagent	Alias	Concentration	Directions	Shelf Life
Sodium Hydroxide		10 N	Weigh 400 g NaOH into ~400 ml DI water in a 1-L volumetric flask. Dilute to volume.	1 year
Nitric Acid		1 N		Manufacturer expiration date or 2 years from receipt
Glacial Acetic Acid		Concentrated		
Hydrochloric Acid		1 N		
TCLP Extraction Fluid #1	TCLP Fluid #1	pH 4.93 \pm 0.05	In 23 L carboy add 20 L of DI water. Add 228 ml glacial HOAc. Add 256 ml 10 N NaOH. Mix well. PH must be 4.93 \pm 0.05.	1 week
TCLP Extraction Fluid #2	TCLP Fluid #2	pH 2.88 \pm 0.05	In 12 L carboy add 10 L DI water. Add 68.4 ml glacial HOAc dilute to volume. pH must be 2.88 \pm 0.05.	1 week
Sulfuric Acid/Nitric Acid	NA	60/40 weight percent mixture	Mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.	6 months
SPLP Extraction Fluid #1	SPLP Fluid #1	pH 4.20 \pm 0.05	Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until the pH is 4.2 \pm 0.05.	1 week
SPLP Extraction Fluid #2	SPLP Fluid #2	pH 5.00 \pm 0.05	Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until the pH is 5.0 \pm 0.05	1 week

Attachment I: Glass Bottle - Blank Rotation Log



Glass Bottle - Blank Rotation Log

Logbook Number: 3905


	Date	Analyst	Bottle																			
			G1	G2	G3	G4	G5	G6	G7	G8	G9	G10	G11	G12	G13	G14	G15	G16	G17	G18	G19	G20
1																						
2																						
3																						
4																						
5																						
6																						
7																						
8																						
9																						
10																						
11																						
12																						
13																						
14																						
15																						
16																						
17																						
18																						
19																						
20																						

Bottle Replacement: Bottles are replaced as a result of damage and/or other issues such as contamination.
 B = Used for Blank
 1 = Used for Sample

Peer Review: _____ Date: _____

Bottle ID	Date Replaced	Reason Replaced

Attachment II: Plastic Bottle - Blank Rotation Log



Plastic Bottle - Blank Rotation Log

Logbook Number: 3906

	Date	Analyst	Bottle																			
			P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12	P13	P14	P15	P16	P17	P18	P19	P20
1																						
2																						
3																						
4																						
5																						
6																						
7																						
8																						
9																						
10																						
11																						
12																						
13																						
14																						
15																						
16																						
17																						
18																						
19																						
20																						

Bottle Replacement: Bottles are replaced as a result of damage and/or other issues such as contamination.

B = Used for Blank
1 = Used for Sample

Peer Review: _____ Date: _____

Bottle ID	Date Replaced	Reason Replaced

Attachment III: ZHE Vessel - Blank Rotation Log

Logbook Number: 3904



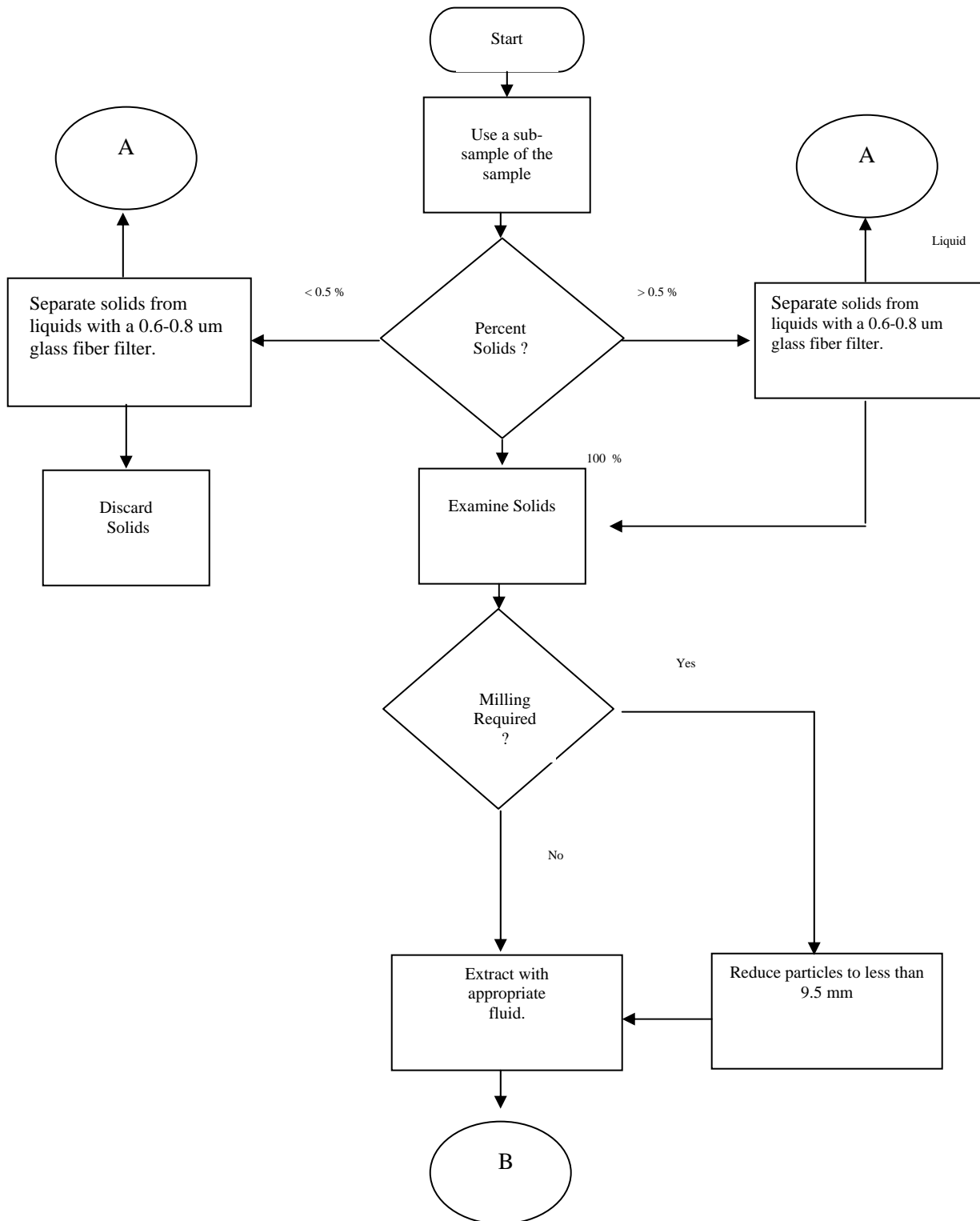
ZHE Vessel - Blank Rotation Log

	Date	Analyst	Vessel										
			A	B	C	D	E	F	G	H	I	J	K
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													

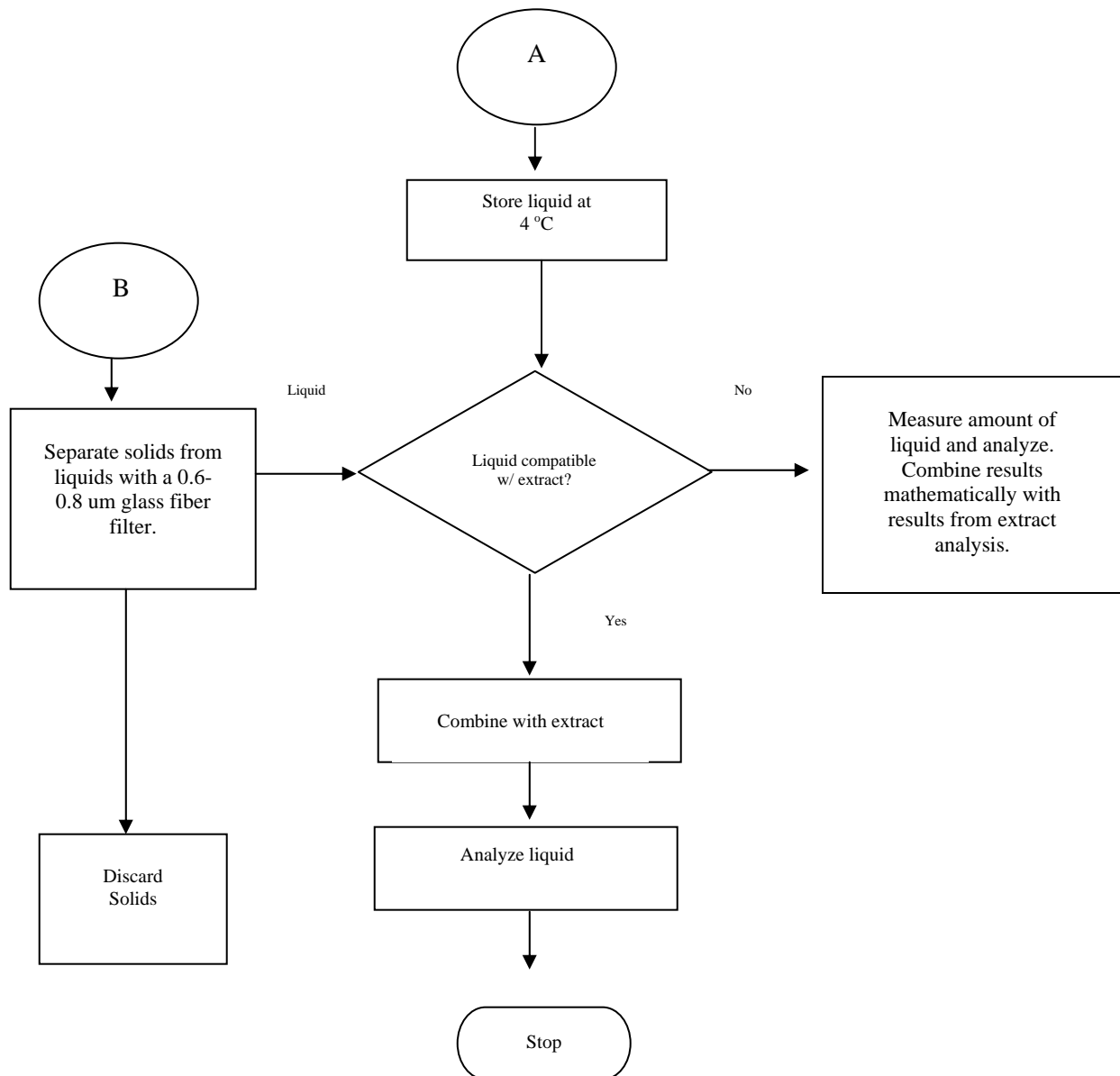
B = Used for Blank
 1 = Used for Sample

Peer Review: _____ Date: _____

Attachment IX: Flow Charts
FLOWCHARTS



FLOWCHARTS



STANDARD OPERATING PROCEDURE

Determination of Trace Metals in Waters and Wastes By Inductively Coupled Plasma Mass Spectroscopy

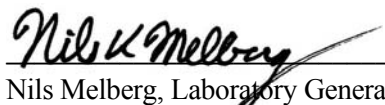
Reference Methods: SW-846 6020, SW-846 6020A and EPA 200.8

SOP NUMBER: S-GB-M-006-REV.07

EFFECTIVE DATE: Date of Final Signature

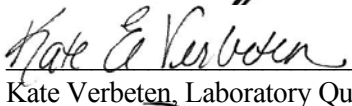
SUPERSEDES: S-GB-M-006-REV.06

APPROVAL



Nils Melberg, Laboratory General Manager

09/14/16
Date



Kate Verbeten, Laboratory Quality Manager

8/19/16
Date



Chad Rusch, Department Manager

08/19/2016
Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature

Title

Date

Signature

Title

Date

Signature

Title

Date

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1. Purpose/Identification of Method

- 1.1 The purpose of this Standard Operating Procedure (SOP) is to provide a consistent format for analyzing samples using an ICPMS. It applies to the Thermo X Series II ICPMS.

2. Summary of Method

- 2.1 Prior to analysis, samples which require total (“acid-leachable”) values must be digested using appropriate sample preparation methods. A number of methods are recommended in SW846, EPA 200.7, and 200.8 for the sample preparation/digestion of various matrices for trace metals analysis by ICPMS. Applicable analytes are listed in Table A.
- 2.2 Methods 6020/6020A/200.8 describe the multi-elemental determination of analytes by ICPMS. The method measures ions produced by a radio frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix as well as isobaric elemental corrections.

3. Scope and Application

- 3.1 Inductively coupled plasma-mass spectrometry (ICPMS) is applicable to the determination of sub-ppb ($\mu\text{g/L}$) concentrations of a large number of elements in biota, aqueous, and solid extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. Dissolved samples are typically acid digested prior to analysis. Acid digestion prior to centrifuging and analysis is required for aqueous and solid samples for which total (acid-leachable) elements are required.
- 3.2 This SOP is applicable to the determination of the analytes listed in Table A, at a minimum. Please see the QA manager for the most current LODs and LOQs for each element and method.
- 3.3 If Methods 6020/6020A/200.8 are used to determine any analytes not listed in Table A, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the matrix to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.
- 3.4 Use of this method is restricted to analysts who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

4. Applicable Matrices

- 4.1 This SOP is applicable to aqueous (dissolved, total, waste) samples.
- 4.2 This SOP is applicable to ASTM, SPLP, and TCLP samples.
- 4.3 This SOP is applicable to solid/soil samples.

4.4 This SOP is applicable to Biota (biological) samples.

5. Limits of Detection and Quantitation

5.1 All current LODs and LOQs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

- 6.1 Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instrument's spectrometer. One way to solve this problem is to measure a different isotope for which there is no interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.
- 6.2 Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. An example includes ClO^+ (mass 51), which interferes with V , and must be corrected by measuring ClO^+ at mass 53. When possible an interference free isotope should be chosen for measurement.
- 6.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.
- 6.4 Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.
- 6.5 It is important to note that matrix matching acid concentrations and compositions between standards, blanks and samples is required and cannot be ignored.
- 6.6 Chromic acid should never be used to clean any container used in ICP-MS analysis.
- 6.7 Borosilicate glass in sample containers can lead to interference of Boron concentrations in samples. This is also true of volumetric flasks, thus when dilution in the flask is complete the standards must be removed as soon as possible from the dilution container and placed into a clean plastic container.

7. Sample Collection, Preservation, Shipment and Storage

- 7.1 All sample containers must be HDPE, glass, or Teflon. The containers are purchased pre-cleaned and documented to be contaminant free.
- 7.2 Aqueous Samples
- 7.2.1 Dissolved samples must be filtered through a 0.45- μ m pore diameter membrane filter at the time of collection or as soon after as practically possible. The laboratory can perform the filtration if the step was not performed in the field. The filtrate is then preserved to a pH<2 with nitric acid, acid not to exceed 2% of the container capacity.
- 7.2.2 Total samples are preserved to a pH<2 with nitric acid, acid not to exceed 2% of the container capacity.
- Note: Aqueous samples that react violently to the addition of acid maybe collected without chemical preservation with proper variances approved by the regulatory authority. The responsibility of requesting this variance lies with the sample collector.**
- Note: Samples may be preserved in the lab. The samples may not be processed until 24 hours after preservation with a pH test of <2, unless otherwise noted on the data. Samples that do not attain a pH<2 or samples that are filtered in the lab must be flagged with P4.**
- 7.3 Solid samples require no chemical preservation.
- 7.4 Tissue (biota) samples are to be shipped at 6°C or less, preferably on dry ice.
- 7.5 Shipments of soil and water samples to the laboratory require thermal preservation in the form of cubed or block ice. At the time of laboratory receipt, proper thermal preservation is checked by measuring the temperature of melt water or when provided the temperature blank. The Pace Analytical acceptable temperature range is 0 to 6°C. All QAPjP and regulatory authority requirements become priority over this requirement.
- 7.6 There are no thermal storage requirements for preserved aqueous samples. Solids samples require storage at 0 to 6°C. Tissue (biota) samples are stored at -10°C or less. . All QAPjP and regulatory authority requirements become priority over this requirement.
- 7.7 Sample Hold Times:
- 7.7.1 The hold time for solid and preserved aqueous (total and dissolved) samples, excluding Hg, is 6 months.
- 7.7.2 The hold time for solid and preserved aqueous (total and dissolved) samples for Hg is 28 days.
- 7.7.3 The hold time for biota samples, excluding Hg, is 6 months after removal from the freezer.
- 7.7.4 The hold time for biota samples for Hg is 28 days after removal from the freezer.

7.8 Samples that are not received according to the SOP are given an appropriate data qualifier.

7.8.1 P4 - Sample field preservation does not meet EPA or method recommendations for this analysis.

7.8.2 C4 - Sample container did not meet EPA or method requirements.

8. Definitions

8.1 Refer to the most current version section 10 of the Pace Quality Manual.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1 All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) should be sufficiently clean for the task objectives.

9.2 See Table B for a summary of Equipment.

9.3 See Table C for a summary of supplies.

10. Reagents and Standards

10.1 Standards are used in the tuning of the instrument through the calibration, calibration verification, sample analysis, and continuing calibration verification. Standard solutions include: instrument tuning solutions, calibration and calibration check standards, ICSA/AB, internal standards, low level check standards (LLC, CRI, or CRDL), and spike standards. **Please see Table D for a list of working standards. Table F lists the directions to making intermediate standards from the stock standards listed in Table E.**

10.1.1 Mass Spectrometer Tuning Solution: A solution containing elements representing all of the mass regions of interest (for example, 10 µg/L of ²³, ²⁴, ²⁵Mg and ²⁰⁶, ²⁰⁷, ²⁰⁸Pb) must be prepared to verify that the mass resolution and mass calibration of the instrument are within the required specifications. This solution is also used to verify that the instrument has reached thermal stability.

10.1.2 Cross Calibration Solution (X Cal): contains 50 ppb of all method analyte elements. This solution is used to calculate the concentration when the detector changes from pulse mode to analog mode. This enables a large linear range while protecting the detector.

10.1.3 Calibration Standards: These are an increasing gradient of concentration for the analytes of interest. These can be made in the laboratory or purchased from commercial suppliers.

10.1.4 Initial Calibration Verification (ICV): The quality control standard is the initial calibration verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard at approximately half (or less than) of the concentration of the high standard used for instrument calibration. An independent standard is defined as a standard composed of the analytes from a source different from those used in the standards for instrument calibration.

- 10.1.5 Reporting Limit Verification Standard (RLVS / CRDL): With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL), or lower, must also be analyzed and meet established acceptance criteria. The RLVS is analyzed prior to any samples being analyzed. It is also analyzed at the end of the analytical sequence when following 6020A or by client request. Additional RLVSs may be analyzed throughout the analytical sequence at the analyst's discretion. These standards are the first calibration point for the analytes of interest. The analysis of this standard demonstrates the instruments ability to report down to the reporting limit with known accuracy.
- 10.1.6 Interference Check Solutions (ICSA and ICSAB): The ICSA and ICSAB are prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. The ICSA and ICSAB are analyzed prior to any samples. They are also analyzed at the end of the analytical sequence when following 6020A or by client request. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interferences such as $^{35}\text{Cl } ^{16}\text{O}$ on ^{51}V . Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various polyatomic isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system to within quality control limits. Table D provides a summary of the ICS-A and ICS-AB solution concentrations used.
- 10.1.7 Continuing Calibration Verification (CCV): This standard is near (or less than) the midpoint of the curve and used to verify that the instrument is still in calibration. This standard is from the same source as the calibration standards.
- 10.2 Reagents: **Please see Table G for a list of reagents.**
- 10.3 All standards, reagents, and spiking solutions are kept at room temperature. Stock standards and reagents can be used until they expire. Refer to the most current version of S-ALL-Q-025 *Standard and Reagent Management and Traceability* for stock standard and reagent expiration rules. Intermediate and working standards are given a six month expiration date. However the expiration date may not extend past the earliest expiration date of any stock standard used to create the solution. The use of any standard or reagent will be terminated if any contamination or problems arise prior to the expiration date.
- 10.4 Log-in of Standards, reagents, and spike solutions are logged as follows:
- 10.4.1 Stock standards have a copy of their certificate of analysis logged into the LIMS (Epic Pro). Please see the SOP T-ALL-IT-010 (most current revision or equivalent).
- 10.4.2 Reagents are logged in the same manner.

11. Calibration and Standardization

- 11.1 The instrument is calibrated daily at a minimum when analyzing samples. High solids and complex matrices can and do result in more frequent calibration.
- 11.2 Calibration Curve: Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required periodically throughout sample analysis as dictated by the results of the continuing calibration verification standards. Calibration consists of a calibration blank and four or five non-zero standards (analyte dependent) analyzed after a calibration blank. The ICPMS software creates a curve based on this data which is linear regression using Absolute Standard Deviation weighting and uses the following equation: $Y=a(x)+b$. In calculating x(concentration) from y(response) the b(intercept) is subtracted. The b represents the intercept of the regression line. The correlation coefficient must be ≥ 0.998 . If the correlation coefficient is not within criteria, the issue must be resolved and the instrument recalibrated with passing criteria prior to analyzing samples.
- 11.3 Calibration Levels: **Please see Table D for directions on making the calibration standards and Table H to see the calibration levels used in the calculations.**
- 11.4 Internal Standards:
- 11.4.1 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. For full mass range scans, a minimum of six internal standards must be used. Procedures described in this SOP for general applications detail the use of six internal standards; ^{45}Sc -CCT, ^{45}Sc -KED, ^{72}Ge , ^{89}Y , ^{115}In , ^{159}Tb , and ^{209}Bi . The interpolation between 2 internal standards is done when a reported element mass is bracketed by two internal standards. **Table I provides a list of acceptable internal standards and their suggested associated elements.**
- 11.4.2 Internal standards must be present in all samples, standards and blanks at identical levels. This is achieved by directly adding the internal standard stock solution (100ppb in 3% HNO_3) to all samples, standards and blanks by on-line addition prior to nebulization using a second channel of the peristaltic pump and a mixing T-connector. The concentration of the internal standard should be sufficiently high that good precision is obtained in the measurement of the isotope used for data correction and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. A final concentration at the nebulizer of approximately 50 ppb will result from the addition of a 100 ppb solution.
- 11.4.3 Due to matrix interferences one or more of the internal standards may not be suitable. It is up to the analyst to recognize and correct the problem by diluting the sample(s) for reanalysis. This procedure may result in some analytes being diluted below the PRL and must be appropriately flagged.
- 11.4.4 During the determination, the software uses the ratio of analyte and internal standard intensities to adjust the final concentration values. Ratios are based on the intensities in the sample vs. the calibration blank intensities.

- 11.4.5 An internal standard without Li must be used if Li is an analyte of interest. If Li is to be used as an internal standard, and Li is an analyte of interest, an isotopically enriched Li standard must be used such that the analyte can be differentiated from the internal standard. (i.e. ^6Li enriched internal standard to report ^7Li in samples.)
- 11.5 Interference Calculations: Interference equations are used to correct for isobaric elemental and polyatomic interferences. All equations can be adjusted if necessary, or added if the analyst determines that any particular correction is insufficient or if an equation is over correcting the data. **Table J shows the recommended Elemental Interference Equations.**
- 11.6 Acquisition Mode:
- | | |
|--------------------|--|
| Points per Mass : | 1 |
| Number Replicates: | 3 |
| Dwell Time: | 10 ms for all elements except Na, As, Se, Kr; 5 ms for Na, and 50 ms for As, Se, Kr. |
- 11.7 Example Peristaltic Pump Program:
- | | |
|--------------------------------------|---------------------|
| Uptake time: | 20 sec ¹ |
| Neb Settle Time: | 20 sec |
| Stabilization Time (KED Changeover): | 40 sec |
- Acquisition masses and Dwell times can be found in Table K.**

12. Procedure

- 12.1 Solubilization and digestion procedures are in the Metals Digestion SOPs (e.g. Methods 200.8, 3010A and 3050B for both Solids and Biota).
- 12.2 Instrument Startup:
- 12.2.1 Verify argon supply and pressure.
 - 12.2.2 Turn on water chiller and exhaust fan.
 - 12.2.3 Ensure that the internal standard solution bottle is adequately full.
 - 12.2.4 Verify contents of auto-sampler rinse port reservoir.
 - 12.2.5 Empty the waste reservoir.
 - 12.2.6 Ignite the plasma and allow at least 25 minutes of warm-up while scanning the mass analyzer. The tuning procedures may then be carried out.
 - 12.2.7 Ensure that all peristaltic pump tubes are in good condition and correctly clamped into the peristaltic pumps. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer are free from extreme pulsations by introducing a bubble into each line and observing its progress.

- 12.3 Mass Tuning: Allow 25 minutes of scanning for the Instrument to achieve thermal stability. Aspirate the 10ppb Tuning Solution by inserting both the sample and internal standard delivery lines into the tune solution, so as not to dilute the tune solution. Run each of the following performance reports, print the passing report, and save the mode by month, day, year (no commas) into the configurations list by type (e.g. Standard Mode09052008, CCT Mode09052008, and CCTKED Mode09052008).

12.3.1 EPA Performance Report (Xt Standard Mode)

12.3.1.1 Mass Resolution (10 Sweeps, 5 Reps)

Acquisition Parameters: Peak width measured at 5% peak maximum.

Dwell Time (mSec): 10

Point Spacing: 0.05amu

Mass Limits: 0.65 – 0.85 amu (Max error 0.10 amu)

Defined Masses: ²⁴Mg, ²⁵Mg, ²⁶Mg, ²⁰⁶Pb, ²⁰⁷Pb, ²⁰⁸Pb

12.3.1.2 Sensitivity and Stability (35 Sweeps, 5 Reps)

Mass: Dwell (mSec): %RSD: Counts

(NA=Not Applicable)

⁵Bkg 500.0: NA: <1

⁷Li 10.0: 2.0 : >60,000

²⁴Mg 10.0 : 2.0 : >10,000

²⁵Mg 10.0 : 2.0 : >10,000

²⁶Mg 10.0 : 2.0 : >10,000

⁵⁹Co 10.0 : 2.0 : >150,000

¹³⁷Ba++ 10.0 : NA : NA

¹¹⁵In 10.0 : 2.0 : >400,000

¹³⁷Ba 10.0 : NA : NA

¹³⁸Ba 10.0 : NA : NA

¹⁴⁰Ce 10.0 : NA : NA

¹⁵⁶CeO 100.0 : NA : NA

²⁰⁶Pb 10.0 : 2.0 : >10,000

²⁰⁷Pb 10.0 : 2.0 : >10,000

²⁰⁸Pb 10.0 : 2.0 : >10,000

²²⁰Bkg 500.0 : NA : <1

²³⁸U 10.0 : 2.0 : >800,000

12.3.1.3 Oxides and Doubly Charged (Ratio Results)

¹³⁷Ba++/¹³⁷Ba: <0.0300

¹⁵⁶CeO/¹⁴⁰Ce: <0.0200

12.3.2 CCT Performance Report (Xt CCT)

Sensitivity and Stability (35 Sweeps, 5 Reps)

Acquisition Parameters: Mass : Dwell (mSec) : %RSD : Counts

⁷Li 10.0: 2.0: >10,000

⁹Be 10.0: 2.0: >2,000

¹¹B 10.0: 2.0: >2,000

12.3.3 KED Performance Report (Xt CCT-KED)

12.3.3.1 Sensitivity and Stability (35 Sweeps, 5 Reps)

Acquisition Parameters: Mass: Dwell (mSec): %RSD: Counts

⁷⁸Se 100.0: NA: <20

¹¹⁵In 10.0: 2.0: >100,000

¹⁴⁰Ce 10.0: NA: NA

¹⁵⁶CeO 50.0: NA: NA

12.3.3.2 Ratio Results

¹⁵⁶CeO/¹⁴⁰Ce <0.0200

12.3.4 Introduce the internal standard and aspirate a new rinse blank for 5-10 minutes to eliminate any carry-over into the calibration blank.

12.3.5 The Xcal solution does not need to be run unless a deviation between the pulse counting and analog counting methods is observed in the spectra. The analog counting will appear to sit above the pulse baseline in an observed spectra and indicates that the cross calibration needs to be performed. Also, an indication of when the Xcal needs to be reset is when the calibration loses its linearity (this will likely occur first with High Resolution Mineral elements like Na and K). Aspirate the Xcal solution in Standard Mode, with both the sample delivery and internal standard lines (so as to not dilute the Xcal solution).

12.3.5.1 Cross Calibration Only. This is done when the deviation between the pulse and analog counting methods is observed (from spectra or from linearity observations) and the minimum counts per second listed in the tuning section are achievable. Run the Instrument Calibration wizard in PlasmaLab when deviation of the analog spectra is observed. The detector cross calibration is selected by default in the wizard. This will reset the detector-gating plateau such that the analog spectra (dashed line in the spectra) will sit directly on top of the pulse baseline (solid line in the spectra).

12.3.5.2 Detector Setup and Cross Calibration. This is done when the minimum counts per second listed in the tune section are not achievable. Thus, the detector dynode value will be adjusted and a new cross calibration will be performed with the new detector voltage setting. Launch the Instrument Calibration wizard in PlasmaLab and select detector set up. The detector cross calibration will be checked by default, also select the detector set up portion in the wizard. The voltage applied to the detector will be set first to achieve acceptable sensitivity followed by a detector cross calibration with the new detector setting.

12.3.5.3 Dead Time Experiment. This experiment must be and is only performed after a detector is replaced. The dead time experiment will identify the delay or “dead time” associated with a particular detector. The dead time is the delay between the pulse measurement and analog measurement when the detector gate is dropped. In the experiment a 1ppb and 10ppb solution of Uranium is run, and the ratios between ^{235}U and ^{238}U are calculated for both concentrations. The values for the ratios are adjusted such that the ratio values are as close to identical as possible and that value (dead time in nanoseconds) is entered into PlasmaLab software via the Advanced Page.

12.3.6 Tune Procedure Summary (Use this order of events, some adjustments may be required after an auto tune)

12.3.6.1 Torch Box Alignment

12.3.6.2 Xt Standard Mode Autotune or X Series II Factory auto tune; Mass Calibration.

12.3.6.3 EPA Performance Report

12.3.6.4 Detector X Calibration (Note: Return to 11.3.6.2 if the Cross Calibration trips a detector set up sequence, excluding the Mass Calibration)

12.3.6.5 XT CCT Mode Auto tune

12.3.6.6 XT CCT Mode Performance Report

12.3.6.7 XT CCT/KED Auto tune

12.3.6.8 XT CCT/KED Performance Report

12.3.6.9 The tunes for passing performance reports are valid for 36 hours.

12.4 Automated Calibration, Quality Control and Sample Analysis:

12.4.1 Prepare calibration standards, blanks, spikes, samples, and QC samples.

Directions to make up the standards can be found in Tables D and F.

12.4.2 Build a sequence in the sequence table and apply the repeat run rules to insert the CCV and CCB for every 10 unknowns. A typical sequence will consist of a calibration blank and calibration points 1 through 6, followed by an ICV, ICB, CRDL, ICSA, ICSAB, CCV, CCB and then batch samples and QC. The repeat rules set above will insert a CCV, CCB combo every 10 analyses and at the end of the sequence.

12.4.3 Ensure the RLVS / CRDL standards, ICSA, and ICS-AB bracket the sample list for analysis by 6020A.

12.4.4 When the sample list and QC checks have been verified for accuracy, queue the experiment to the Technician Queue. Each experiment will require a unique code (e.g. 0905208A, 0905208B, etc.).

12.4.5 Expected values and limits for ICV, CCV, ICS-A, and ICS-AB **can be found in Table L.**

13. Quality Control: are three levels of quality control utilized in this SOP. They consist of Method QC, Instrument QC, and Prep/Batch QC.

13.1 **Method QC** consists of the instrument detection limits, linear ranges, method detection limits, the lower limit quantitation check, and demonstrations of capability. This QC must be completed prior to analyzing any samples.

13.1.1 Instrument Detection limits (IDLs): IDLs in µg/L are estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with ten consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined quarterly.

13.1.2 Linear Dynamic Ranges (LDR): The LDR for each element is determined by analyzing a standard series ending at a concentration expected to be near the high end of the instruments range. The result must be within $\pm 10\%$ of the expected concentration, or a lesser concentration tested until acceptable results are obtained. The element may be reported to 90% of the highest passing LDR standard tested. After the initial determination the high point is verified every 6 months (semi-annually) using the same $\pm 10\%$ of the true value.

13.1.3 Method Detection Limits (MDLs): Method detection limits, at a minimum, are obtained by multiplying 3.143 by the standard deviation of seven spikes. The spikes went through the digestion procedures and were spiked at a concentration suspected to be within a factor of ten greater than the MDL. The most current version of the MDL SOP, S-ALL-Q-004, must be followed when determining MDLs. Note: The MDL determined by the MDL study is the theoretical MDL. The MDL used for reporting purposes may be higher than the theoretical MDL.

13.1.4 Lower Limit Quantitation Check (LLQC): The LLQC is a standard at the reporting limit concentration that has gone through all the preparation procedures. It has control limits of $\pm 30\%$ of the true value. It is analyzed to establish the lower limit of quantitation, verified annually, and whenever new limits are established. If an element repeatedly does not meet criteria at a certain level, it should be re-evaluated to determine if that level is realistic.

13.1.5 Initial Demonstration of Capability (IDC): An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

- 13.2 **Instrument QC:** Prior to the analysis of samples, and in some cases during the analysis run, the following quality control must be generated and within limits: Performance Report for each mode of operation generated daily (Up to 36 hours since previous), Internal Standards, Interference Correction Solutions A and B (ICSA and ICSAB), Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), RLVS / CRDL standards, and Initial and Continuing Calibration Blanks (ICB and CCB).
- 13.2.1 The intensities of all internal standards must be monitored for every analysis. When the intensity of any required internal standard fails to fall between 70 and 125 percent of the intensity of that internal standard in the blank of the calibration, one of the following procedures is followed. The sample is diluted appropriately and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the required internal standard intensities fall within the prescribed window. If significant deviation cannot be overcome by dilution of the sample in the same analytical run then recalibration is required. Sudden increases in internal standard recoveries of 10 or more percent may also indicate that dilutions are necessary. The presence of high levels of dissolved solids in samples can result in instrument drift. A sequence with high solids samples will have recoveries drift down. Should the internal standards drift outside of the acceptance range; these samples will need to be re-analyzed at a dilution. If a subsequent batch is clean (samples with low dissolved solids present) the instrument may see an increase in signal and internal standard recovery. Unless the internal standards are actually present in the samples, these can be re-analyzed at the same dilution after a passing recalibration.
- 13.2.2 The ICSA and ICSAB solution are used to verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections. They are analyzed after the CRDL, prior to the analysis of any samples. For 6020 and 200.8, additional ICSA and ICSABs are not analyzed, as sequences do not reach 12 or more hours in duration. For 6020A and by client request, the ICSA and ICSAB will also be analyzed at the end of the analytical sequence. The analyst should be aware that precipitation from solution AB may occur with some elements, specifically silver. The control limits for the elements in the ICSA and ICSAB solutions is 80 to 120% of the expected recovery for elements with concentrations within the linear range. Elements that are not spiked must have a measurement lower than the LOQ. If an element of interest does not meet these limits, then the problem must be corrected, the instrument recalibrated, and the run reanalyzed.
- 13.2.3 The ICV is analyzed to check the accuracy of the curve. It must be evaluated after each calibration and before any samples are analyzed. The ICV should be at or near (or lower than) the midpoint of the calibration curve, derived from a source independent of the calibration standards, and must quantitate within $\pm 10\%$ of the expected value. If these limits are not met for an element of interest, the problem must be corrected, the instrument recalibrated, and the run reanalyzed. If the ICV fails high and the samples are non-detects, then they may be reported.

- 13.2.4 Reporting Limit Verification Standard (RLVS / CRDL): With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) or lower must also be analyzed and meet established acceptance criteria. The RLVS is analyzed prior to any samples being analyzed. For 6020A and by client request it will also be analyzed at the end of the analytical sequence. Additional RLVSs may be analyzed throughout the analytical sequence at the analyst's discretion. The limits for the RLVS are $\pm 30\%$ of the true concentration. These standards are the first calibration standard for the analytes of interest. The analysis of this standard demonstrates the instruments ability to quantify down to the reporting limit with known accuracy. If an element fails the CRDL, but the CCV passes and the samples are greater than the CCV concentration, then the sample data may be reported. All others must be re-analyzed under passing criteria.
- 13.2.5 The CCV is analyzed to check for calibration drift. The CCV is run prior to any samples, after every 10 samples and again at the end of samples. It must quantitate within 10% of the expected value. Any sample analyzed under out-of-control calibration must be reanalyzed, following the successful re-calibration of the instrument. If the CCV fails high and the samples are non-detects, then they may be reported. All others must be re-analyzed under passing criteria.
- 13.2.6 The ICB is analyzed to check the accuracy of the curve. The CCBs are analyzed to check for calibration drift. In the absence of project specific reporting limits, the results of the calibration blanks must be less than the Reporting Limit. The ICB is run after the ICV and the CCBs are run after the CCVs. If the ICB or CCB fails high for an analyte and the samples are non-detects or are greater than 10x the blank value, then they may be reported. All others must be re-analyzed under passing criteria.
- 13.3 A batch will consist of 20 or fewer samples. **Batch Quality Control** will include a Method Blank (MB), Chicken Blank (CB) (Biota only), Laboratory Control Spike (LCS), Matrix Spike (MS), Matrix Spike Duplicate (MSD), Post Digestion Spike (PDS), and a Serial Dilution (SD). It may also include a Laboratory Control Spike Duplicate (LCSD) and/or Standard Reference Material (SRM)
- 13.3.1 The Method Blank is used to verify that interferences caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized. The method blank is processed through all clean-ups, etc., which were performed on the samples in the batch. For a method blank to be acceptable, in the absence of project specific criteria, the concentration shall not be higher than the highest of the following: The reporting limit, or ten percent of the regulatory limit of concern for that analyte, or ten percent of the measured concentration in a particular sample of interest. Each sample in the batch is assessed against the above criteria to determine if the sample results are acceptable. Any sample associated with an unacceptable blank is either flagged, re-prepped for analysis, or if re-prepping is not an alternative, the results are reported with the appropriate data qualifying codes.

13.3.2 The Chicken Blank (CB) must be prepared with every biota batch, and is processed through all clean-ups, etc., which were performed on the samples. It consists of a control matrix (ground chicken) to verify the interferences in the biological tissue is known and minimized. The CB will contain detectable amounts of elements such as K, Ca, Na, Mg, and P etc., and is used to ensure acceptable performance of the laboratory control spike. Any detections in the CB greater than the 40CFR Part 136 statistically derived MDL is subtracted from the LCS/LCSD before recovery limits are evaluated.

13.3.3 A laboratory control sample (LCS) consists of a control matrix, which has been spiked, with the analytes(s) of interest or compounds representative of those analytes. Laboratory Control Samples are analyzed at a minimum of 1 per batch of 20 or fewer samples or preparation method. Results of the LCS are expressed in terms of percent recovery, and are used to determine batch acceptance. Acceptance limits for 6020 and 6020A are 80 to 120%. Acceptance limits for 200.8 are 85 to 115% of the expected recovery. If these limits are not met with the instrument in control, then the entire batch will be re-digested and re-analyzed.

An LCS Duplicate may be analyzed to evaluate laboratory precision. The LCSD must also meet the criteria for the LCS. The Relative Percent Difference (RPD) will be calculated between the LCS and LCSD. The RPD is calculated as outlined below:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2) / 2} \times 100$$

Where:

RPD = relative percent difference.

D₁ = first sample value.

D₂ = second sample value (duplicate)

The control limit for RPD is exceed 20%, or is based on laboratory generated data and not to exceed 20%. If outside this limit, all associated results are given a R1 data qualifier. Data generated with LCS samples that fall outside the established acceptance criteria are judged to be out-of-control. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

For the biota matrix, any detections greater than the MDL must be subtracted from the LCS/LCSD on-instrument value before calculating the recovery limits.

NOTE: In the event where adequate sample is not supplied by the client to perform a Matrix Spike/ Matrix Spike Duplicate, the LCS and duplicate can lend insight on the precision of the analysis.

- 13.3.4 Matrix spikes (MS and MSD) are performed to evaluate the effect of the sample matrix upon analytical methodology. A separate aliquot of sample is spiked with the analyte of interest and analyzed with the sample. For 6020 and 6020A an MS and MSD are performed at a minimum frequency of 5% per batch. One pair in 20 samples, per matrix type, per sample preparation method and are performed more frequently where regulations require. For 200.8, an MS/MSD pair, are performed at a 10% frequency per batch. Matrix spike recoveries are evaluated against in-house control limits. The recovery must not exceed 75 to 125% of the expected recovery. If outside this recovery, the parent is flagged with an appropriate data qualifier. If the recovery of an analyte is outside this range but the spike level is not at least 25% the background concentration in the parent sample, the data is flagged with an appropriate data qualifier. The RPD between the MS and MSD must be less than 20 %. If outside this limit, the parent is given an appropriate data qualifier. The parent sample for the MS/MSD is chosen at random unless specified by a client. Poor performance in a matrix spike generally indicates a problem with the sample composition, and not the laboratory analysis, and results are used to assist in data assessment. A matrix effect is indicated if the LCS data are within acceptance criteria but the matrix spike data exceed the acceptance criteria. Prior to calculating recovery, the parent sample concentration (results < Reporting MDL = 0) is subtracted from the spike aliquot concentrations.
- 13.3.5 The Post Digestion Spike (PDS) is run to verify matrix interferences. A spike is added to a portion of a prepared sample, or its dilution and must be recovered to within 80 to 120 percent of the known value. If the spike is not recovered within the specified limits with the instrument in control, then the sample has a confirmed matrix effect. Dilute the parent and re-spike the diluted sample until the PDS recovers within acceptance criteria.
- 13.3.6 The Serial Dilution (SD) is run to check for matrix interferences. If the analyte concentration is within the linear dynamic range (LDR) of the instrument and sufficiently high (minimally, a factor of at least 10 times greater than the lower limit of quantitation for the diluted sample, 50 times the RLVS standard for the parent sample, or by project/client specific criteria) an analysis of a fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination. If these limits are not met then an interference effect must be suspected and the data qualified with an SD data qualifier. If the analytes of interest are greater than the LDR in the parent sample, the sample can be diluted and an SDL done off of the dilution.
- 13.3.7 The type of SRM is typically prepped and analyzed upon client request. It is a sample of known concentration chosen to resemble the matrix being analyzed.

14. Data Analysis and Calculations

- 14.1 The Plasma Lab software performs all calculations necessary to convert raw counts per second data into quantitative concentration results.
- 14.2 Consideration should be given to the interference equations that are in the method. As a result of changing tune parameters over time, the equations may require adjustment periodically to ensure they are not over compensating or under compensating for the polyatomic or isobaric interferences. This is especially true of ^{52}Cr and ^{51}V as the CCT-KED tune can vary slightly from day to day operation. This variation (primarily in the add gas for the collision cell) will change the counts per second acquired for Cl and ClO ions. This can make the counts per second in the calibration blank for these isotopes artificially high or low (**See Table J**). In the case of Lead, quantitation is based on the sum of isotopes 206, 207, and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.
- 14.3 See the QA Manager for Isotopes and the respective laboratory specific method detection limits and reporting limits.
- 14.4 Aqueous Sample Calculation:

$$\frac{\text{Raw Data result } (\mu\text{g/L}) * \text{DF} * V_F}{V_I} = \text{Final Result } (\mu\text{g/L})$$

Where:

DF = Dilution Factor

V_F = Final Volume (L)

V_I = Initial Sample Volume (L)

- 14.5 Soil Sample Calculation:

$$\frac{\text{Raw Data result } (\mu\text{g/L}) * \text{DF} * V_F}{W_S * \%S} = \text{Final Result (mg/kg dry weight)}$$

Where:

DF = Dilution Factor

V_F = Final Volume (L)

V_I = Initial Sample Volume (L)

W_S = Sample weight (grams)

$\%S$ = Percent solids/ 100

Example: For a sample that is 97.6% solid use 0.976

14.6 Biota Sample Calculation:

$$\frac{\text{Raw Data result } (\mu\text{g/L}) * \text{DF} * V_F}{W_S} = \text{Final Result (mg/kg)}$$

Where:

DF = Dilution Factor

V_F = Final Volume (L)

V_I = Initial Sample Volume (L)

W_S = Sample weight (grams)

NOTE: Results for biological samples are routinely reported on an “as is” or wet weight basis. Dry weight correction is available on request when sufficient sample has been provided.

14.7 Hardness as CaCO_3 in mg/L = $2.497 * [\text{Ca}_{\text{in mg/L}}] + 4.118 * [\text{Mg}_{\text{in mg/L}}]$

14.8 Silica (SiO_2) ($\mu\text{g/L}$) = Silicon (Si) ($\mu\text{g/L}$) * DF * 60.09 amu (SiO_2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

15. Data Assessment and Acceptance Criteria for Quality Control Measures

Analytical Method Criteria⇒ Data Assessment Measure ⇓	Frequency	Acceptance Criteria
MB	One per batch, not to exceed 20 samples.	< LOD < LOQ
LCS/LCSD	One LCS per batch, not to exceed 20 samples. LCSD performed by client request or if insufficient sample volume for MS/MSD.	6020: ±20% recovery of the true value. 200.8: ±15% recovery of the true value. ≤20% RPD
MS/MSD	200.8 - one pair at 10% sample frequency. 6020/6020A - one pair at 5% sample frequency.	±25% recovery of the true value. ≤20% RPD
PDS	Once per batch of 20 or fewer samples.	±20% recovery of the true value.
SD	Once per batch of 20 or fewer samples.	±10% RPD referenced to the parent
DUP	By client request.	≤20% RPD
SRM	One per batch of 20 or fewer biota samples. Additional SRMs can be performed by client request.	Reference only. Client specific acceptance criteria.
Initial Calibration	Once per day at minimum.	≥0.998 correlation coefficient
ICV	Once right after calibration.	±10% recovery of the true value.
ICB	Once right after ICV.	<RL
ICSA	Prior to any samples, typically after the CRDL.	±20% recovery of the true value. <LOQ for non-spiked elements
ICSAB	After the ICSA.	±20% recovery of the true value.
CRDL	Prior to samples being analyzed, typically after the ICB.	±30% recovery of the true value.
CCV	Prior to any samples. Bracket every 10 or fewer samples. At the end of the sequence.	±10% recovery of the true value.
CCB	Right after CCVs.	< RL

16. Corrective Actions for Out-of-Control Data

Analytical Method Criteria⇒ Data Assessment Measure ↓	If acceptance criteria are not achieved ⇒
MB	• 1
LCS/LCSD	• 2
MS/MSD	• 3
PS	• 4
SDL	• 5
DUP	• 6
SRM	• 7
Initial Calibration	• 8
ICV	• 9
ICB	• 10
ICSA	• 11
ICSAB	• 12
CRDL	• 13
CCV	• 14
CCB	• 15

1. If not <LOQ, verify by second analysis. If second analysis confirms contamination for target analyte at or greater than the LOQ, re-digest sample batch and batch QC provided sufficient sample volume remains. If insufficient sample volume remains, consult with project manager and client on how to proceed. For MB detections greater than or equal to the LOD, but less than the LOQ, qualify applicable sample results. For negative measurements more negative than the LOD, applicable data is given the following data qualifier: "Analyte was measured in the associated method blank at a concentration of -#.# units."
* For positive MB failures, samples that are non-detection need not be qualified. In addition, samples that are greater than 10 times the MB detection need not be qualified.
* For negative MB failures samples that are greater than 10 times the MB detection need not be qualified.
2. Verify failure by second analysis. If second analysis confirms LCS (LCSD) failure, re-digest sample batch and batch QC provided sufficient sample volume remains. If insufficient sample volume remains, consult with project manager and client on how to proceed.
3. If the parent, MS, or MSD is greater than the reportable linear dynamic range, dilute and reanalyze the parent, MS, and MSD. If the concentration of the spike is less than 25% of the concentration of the parent the MS and MSD recoveries are not evaluated. Any failures resulting from this are qualified appropriately. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately qualify the parent sample if either the MS and/or MSD fail accuracy. If the MS and MSD fail precision control limits flag the parent with the appropriate precision data qualifier.
4. If the spike is not recovered within the specified limits with the instrument in control, dilute the parent and re-spike the diluted sample until the PS recovers within acceptance criteria.
5. If these limits are not met then an interference effect must be suspected and the data qualified with an appropriate data qualifier. If the analytes of interest are greater than the LDR in the parent sample, the sample can be diluted and an SDL done off of the dilution.
6. If the DUP fails precision control limits flag the parent with the appropriate precision data qualifier.
7. For Biota analysis only. Outside of client specific criteria, the SRM is digested and analyzed only to demonstrate analyte recovery in a standard reference material. Client specific action may also exist.

8. Correct the issue and recalibrate.
9. Verify failure by second analysis. If second analysis confirms ICB failure, correct the issue and recalibrate the instrument. No data may be reported unless there is a passing ICB for that target element. If verification passes criteria, then proceed with the analytical sequence.
10. Verify failure by second analysis. If second analysis confirms ICB failure, correct the issue and recalibrate the instrument. No data may be reported unless there is a passing ICB for that target element. Exceptions; if the failure is biased high and the sample is a non-detection, or the sample concentration is greater than 10 times the detection in the ICB for that target element.
11. Verify failure by second analysis. If second analysis confirms ICSA failure, the system is out of control. Correct the issue and recalibrate the instrument. No data may be reported unless there is a passing ICSA for that target element.
12. Verify failure by second analysis. If second analysis confirms ICSAB failure, the system is out of control. Correct the issue and recalibrate the instrument. No data may be reported unless there is a passing ICSAB for that target element.
13. Verify failure by second analysis. If second analysis confirms CRDL failure, the system is out of control. Correct the issue and recalibrate the instrument. No data may be reported unless there is a passing CRDL for that target element.
14. Verify failure by second analysis. If second analysis confirms CCV failure, correct the issue and recalibrate the instrument. On unattended sequence, CCVs may fail and then pass later in the sequence, but no data may be reported unless bracketed by passing CCVs for that target element. Exceptions; if the sample element concentration is greater than the CCV concentration and the bracketing CCVs are within control, that sample data may be reported.
15. Verify failure by second analysis. If second analysis confirms CCB failure, correct the issue and recalibrate the instrument. On unattended sequence, CCBs may fail and then pass later in the sequence, but no data may be reported unless bracketed by passing CCBs. Exceptions; if the failure is biased high and the sample is a non-detection, or the sample concentration is greater than 10 times the detection in the CCB for that target element.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

See section 16 Corrective Actions for Out-of-Control Data

18. Method Performance

- 18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.
- 18.2 The analyst must read and understand this procedure with written documentation maintained in his/her training file.
- 18.3 An initial demonstration of capability (IDC) must be performed per the most recent version of S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager. A continuing demonstration of capability (CDOC) must be performed annually.
- 18.4 At a minimum, the 40CFR part 136 appendix b study must be performed every year per the most recent version of S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement). This is to be done for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.
- 18.5 Periodic performance evaluation (PE) samples are analyzed per the most recent version of S-GB-Q-021 *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office. These are performed twice a year per matrix.

19. Method Modifications

- 19.1 The digestion procedures are based on, but differ somewhat from 200.8, 3010A, and 3050B. Pace Wisconsin has conducted temperature, time, and side by side studies to validate the digestions utilized. Pace Wisconsin digestions are typically more aggressive with higher acid concentrations than the methods listed.
- 19.2 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- 19.3 Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- 19.4 When there is insufficient volume provided by the client for the method specified matrix spike/matrix spike duplicate (MS/MSD), a laboratory control spike duplicate will be analyzed to demonstrate precision criteria. Laboratory batches will be qualified with the appropriate “M5” data qualifier. When performing this analysis on paint chip samples, a MS/MSD will not be completed on the samples due to high levels of elements present in the native sample.
- 19.5 Blank limits generated from the IDL, as specified in 6020, are unrealistically low. The more recent 6020A limit (LOQ) is used.

20. Instrument/Equipment Maintenance

- 20.1 See Thermo X Series II operator’s manual for information.

21. Troubleshooting

- 21.1 See Thermo X Series II operator’s manual for information.

22. Safety

- 22.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.
- 22.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training
- 22.3 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the MSDS sheets for all chemicals and reagents they use for this procedure and the location of the MSDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

23. Waste Management

- 23.1 Excess reagents samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

24. Pollution Prevention

- 24.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

25. References

- 25.1 PASI Quality Manual, most current revision or replacement.
- 25.2 The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version
- 25.3 USEPA, SW-846, Method 6020A “Inductively Coupled Plasma – Mass Spectrometry”, February 2007.
- 25.4 USEPA, SW-846, Method 3010A, “Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry”, December 1996.
- 25.5 USEPA, SW-846, Method 3050B, “Acid Digestion of Sediments, Sludges, and Soils”, December 1996.
- 25.6 USEPA, 200.8 Revision 5.4, “Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry”, 1994.

26. Tables, Diagrams, Flowcharts and Validation Data

Flow Chart A -	ICPMS DETERMINATIVE METHOD
Flow Chart B -	APPLICABLE ANALYTESMDL or LOD FLAGGING and REPORTING
Flow Chart C -	EQUIPMENTSMDL or PSRL FLAGGING and REPORTING
Table A -	APPLICABLE ANALYTES
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Table C -	SUPPLIES
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Table E -	STOCK STANDARDS
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Table G -	REAGENTS
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27. Revisions

Document Number	Reason for Change	Date
S-GB-M-006-Rev.06	Throughout Document: Update temperature range to 0-6 and fixed formatting errors.. Section 19: Added method modifications for MS/MSD use. Section 25: Added PASI QM and TNI references. Table J: Updated interference equations for ⁹⁰ Zr..	20Nov2014
S-GB-M-006-Rev.07	Section 3.2, 5.1: Changed from MDL/RL to LOD/LOQ. Section 4: Added leach matrices. Section 7: Added Tissue shipping requirements and container qualifiers. Section 11.1: Added. Section 11.2: Updated with CAR. Section 11.4: Updated IS information, 11.4.3: Removed Note. Section 12.3.6.9: Added. Section 12.4.2: Updated with typical analytical sequence. Section 13: Minor language changes throughout section. Table E: ICSA vendor changed to Organic Ventures. Table G: Added 2 year expiration date. Table J: Updated interference equations.	19Aug2016

Flowchart A
ICPMS DETERMINATIVE METHOD

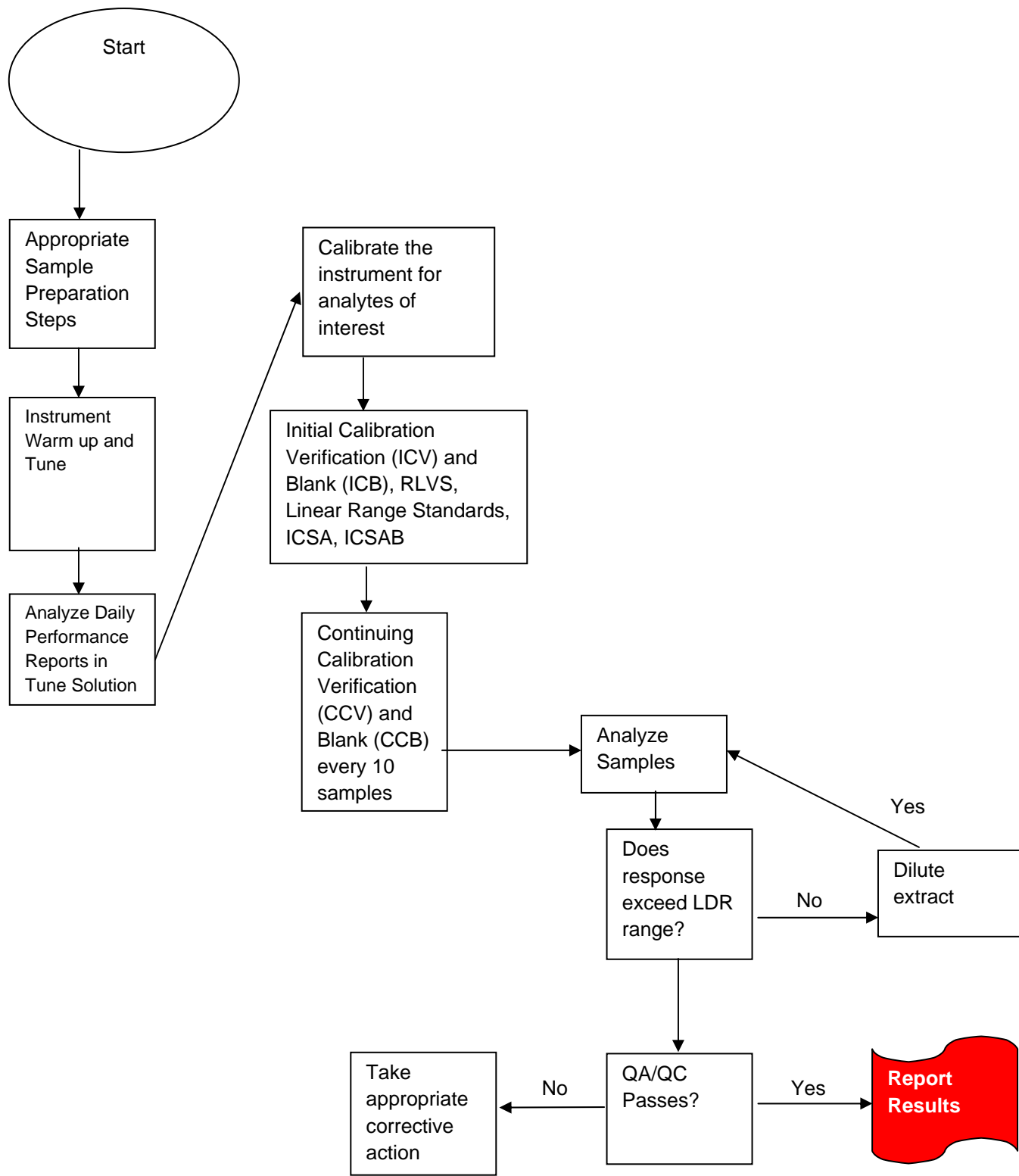


Table A: APPLICABLE ANALYTES

Isotope	Element	Symbol	Chemical Abstracts Service Registry Number (CASRN)
27	Aluminum	Al	7429-90-5
121	Antimony	Sb	7440-36-0
75	Arsenic	As	7440-38-2
137	Barium	Ba	7440-39-3
9	Beryllium	Be	7440-41-7
11	Boron	B	7440-42-8
111	Cadmium	Cd	7440-43-9
43	Calcium	Ca	7440-70-29
52	Chromium	Cr	7440-47-3
59	Cobalt	Co	7440-48-4
65	Copper	Cu	7440-50-8
57	Iron	Fe	7439-89-6
7	Lithium	Li	7439-93-2
208	Lead	Pb	7439-92-1
24	Magnesium	Mg	7439-95-4
55	Manganese	Mn	7439-96-5
201	Mercury	Hg	7439-97-6
95	Molybdenum	Mo	7439-98-7
60	Nickel	Ni	7440-02-0
105	Palladium	Pd	7440-06-4
31	Phosphorous	P	7723-14-0
195	Platinum	Pt	7440-05-3
39	Potassium	K	7440-09-7
82	Selenium	Se	7782-49-2
28	Silicon	Si	7440-21-3
107	Silver	Ag	7440-22-4
23	Sodium	Na	7440-23-5
88	Strontium	Sr	7440-24-6
205	Thallium	Tl	7440-28-0
118	Tin	Sn	7440-31-5
47	Titanium	Ti	7440-32-6
238	Uranium	U	7440-61-1
51	Vanadium	V	7440-62-2
66	Zinc	Zn	7440-66-6
90	Zirconium	Zr	7440-67-7

Table B: EQUIPMENT

Equipment	Manufacturer	Model(#)	Serial #	Model(#)	Serial #
Thermo Xseries 2 ICPMS	ThermoFisher Scientific	XSeries 2	01301	XSeries 2	01780C
Rough Pump	Edwards	E2 M28	006703876	E2 M28	109437330
Computer	Lenova	ThinkCentre	1S3133A3UMJGPFVE	ThinkCentre	00186046357407
Autosampler	CETAC	ASX520	101016A520	ASX520	0610120A520
Refrigerated Recirculator	NESLAB	Merlin M75	108009024	ThermoFlex 2500	110208094
Uninterruptible Power Supply (UPS)	Toshiba	1600 EP Series UPS	080400330	1600 EP Series UPS	100604213
Hot Block	Environmental Express	SC100	526CEC0714	SC100	526CEC0714
Analytical Balance	Denver Instrument	XE-310	718860	XE-310	718860

Note: Equivalent substitutes may be used. Examples include Analytical West for the nebulizer, torch, and the screen/bonnet. Also, Spectron as a substitute for the cones.

Table C: SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #*
Sample Per-Pump Tubing	Analytical West	Analytical West	PT-2100P
Internal Standard Per-Pump Tubing	Analytical West	Analytical West	PT-2100P
Waste Per-Pump Tubing	Analytical West	Analytical West	PT-2160P
Quartz T Connector	Glass Expansion	Glass Expansion	60-808-1185
Concentric Nebulizer	Thermo Electron Corp.	Thermo Electron Corp.	4600294-03
Quartz Torch	Thermo Electron Corp.	Thermo Electron Corp.	3601145
Screen and Bonnet	Thermo Electron Corp.	Thermo Electron Corp.	3601219
Ni Sample Cone	Thermo Electron Corp.	Thermo Electron Corp.	3600812
Ni Skimmer Cone	Thermo Electron Corp.	Thermo Electron Corp.	3600811
Graphite Sample Cone Seal	Thermo Electron Corp.	Thermo Electron Corp.	3004382
15 mL Polypropylene Test Tubes	Fisher Scientific	Fisher Scientific	14-956-7E
50-mL Disposable Digestion Cups	Environmental Express	Environmental Express	SC475
Calibrated Pipette 1000 µL	Eppendorf	Fisher Scientific	21-371-13
Calibrated Pipette 10-50 µL	Thermo	Thermo	21-377-193
Calibrated Pipette 100-1000 µL	Eppendorf	Fisher Scientific	05-402-50
Calibrated Pipette 1000-5000 µL	Eppendorf	Fisher Scientific	05-402-91
Trace Metal Grade Pipette Tips 1 mL	Eppendorf	Fisher Scientific	21-372
Trace Metal Grade Pipette Tips 5 mL	Eppendorf	Fisher Scientific	21-381-198
N-DEX Nitrile Gloves	Best	Fisher Scientific	6005 PFM
pH Indicator Sticks	Whatman	MG Scientific	P114-26

Note: Equivalent substitutes may be used.

**Table D:
WORKING STANDARDS**

Standard	Stock or Intermediate Standard	Concentration	Elements	Amount Used	Final Volume (W/ Diluent)	Final Concentration	Elements
Cross Calibration (XCal)	ICP-MS-68A Solution A	10 ppm Li 20ppm	Al, As, Ba, Be, Bi, B, Cd, Ca, Ce, Cs, Cr, Co, Cu, Dy, Er, Eu, Gd, Ga, Ho, In, Fe, La, Pb, Li, Lu, Mg, Mn, Nd, Ni, P, K, Pr, Re, Rb, Sm, Sc, Se, Na, Sr, Tb, Tl, Th, Tm, U, V, Yb, Y, Zn	0.5 mL	100 mL	50 ppb Li 100ppb	All Elements except Li
	ICP-MS-68A Solution B		0.5 mL				
	Li XCAL Int		0.25 mL				
Tune Solution	Tune Int	1 ppm	Mg, Bi, Ge, In, Sc, Tb, Y, Ba, B, Be, Ce, Co, Li, Ni, Pb, U	5 mL	500 mL	10 ppb	All Elements
ICB	Same as Cal. Level 0						
CCB	Same as Cal. Level 0						
Calibration Level 1 and Diluent/Blank Solution	Cal 0	---	----	-----	50 mL	-----	-----
	Diluent / Blank	----	----	----	1000 mL	----	-----
Calibration Level 1 and RLVS as applicable (See Table H)	Hg Int	1 ppm	Hg	0.010 mL	50 mL	0.2 ppb	Hg
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,	0.04		1 ppb	All Elements (not Ag, Si, P)
		62.5 ppm	P (from two sources)			0.5 ppb	Ag
		0.625 ppm	Ag			50 ppb	Si, P
		62.5 ppm	Si (from two sources)			250 ppb	Al, Ca, Fe, K, Mg, Na
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	0.025			
Calibration Level 2 and RLVS as applicable (See Table H)	Hg Int	1 ppm	Hg	0.025 mL	50 mL	0.5 ppb	Hg
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,	0.2 mL		5 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,
		62.5 ppm	P (from two sources)			2.5 ppb	Ag
		0.625ppm	Ag			250 ppb	Si, P
		62.5 ppm	Si (from two sources)			500 ppb	Al, Ca, Fe, K, Mg, Na
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	0.025 mL			
Calibration Level 3	Hg Int	1 ppm		0.05 mL	50 mL	1 ppb	Hg
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,	2 mL		50 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,
		62.5 ppm	P (from two sources)			25 ppb	Ag
		0.625 ppm	Ag			2500 ppb	Si, P
		62.5 ppm	Si (from two sources)			2,500 ppb	Al, Ca, Fe, K, Mg, Na
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	0.25 mL			

Table D:
WORKING STANDARDS continued

Standard	Stock or Intermediate Standard	Concentration	Elements	Amount Used	Final Volume (W/ Diluent)	Final Concentration	Elements
Calibration Level 4	Hg Int	1 ppm	Hg	0.5 mL	50 mL	10 ppb	Hg
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,	10 mL		250 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,
		62.5 ppm	P (from two sources)			125 ppb	Ag
		0.625 ppm	Ag			12,500 ppb	Si, P
		62.5ppm	Si (from two sources)			12,500 ppb	Si, P
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	1.25 mL		12,500 ppb	Al, Ca, Fe, K, Mg, Na
Calibration Level 5	Hg Int	1 ppm	Hg	1.25 mL	50 mL	25 ppb	Hg
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,	20 mL		500 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,
		62.5 ppm	P (from two sources)			250 ppb	Ag
		0.625 ppm	Ag			25,000 ppb	Si, P
		62.5 ppm	Si (from two sources)			25,000 ppb	Si, P
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	2.5 mL		25,000 ppb	Al, Ca, Fe, K, Mg, Na
CCV	Hg Int	1 ppm	Hg	0.25 mL	50 mL	5 ppb	Hg
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr	4 mL		100 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,
		62.5 ppm	P (from two sources)			50 ppb	Ag
		0.625 ppm	Ag			5,000 ppb	Si, P
		62.5 ppm	Si (from two sources)			5,000 ppb	Si, P
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	0.5 mL		5,000 ppb	Al, Ca, Fe, K, Mg, Na
ICV	U Stock	1,000 ppm	U	0.022 mL	200 mL	110 ppb	U
	Pd Stock	1,000 ppm	Pd	0.022 mL		110 ppb	Pd
	Pt Stock	1,000 ppm	Pt	0.022mL		110 ppb	Pt
	PA-STD-1B	200 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn	0.110 mL		110 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn
	PA-STD-2B	1,000 ppm	Si, P (from two sources)	1.10 mL		5,500 ppb	Si, P
		200 ppm	B, Mo, Sb, Sn, Ti, Zr	0.110 mL		110 ppb	B, Mo, Sb, Sn, Ti, Zr
		100 ppm	Ag			55 ppb	Ag
	PA-STD-3B	2,000 ppm	Al, Ca, Fe, K, Mg, Na	0.550 mL		5,500 ppb	Al, Ca, Fe, K, Mg, Na
Hg ICV Int	10 ppm	Hg	0.08 mL	4 ppb	Hg		
ICSA	4400-061025MB01 Pace	500µg/mL	Al, Ca, Fe, K, Mg, Na, P, S,	10 mL	100 mL	50,000 ppb	Al, Ca, Fe, K, Mg, Na, S, P
		1,000µg/mL	C			100,000 ppb	C
		3,600µg/mL	Cl			360,000 ppb	Cl
		10µg/mL	Mo, Ti			1,000 ppb	Mo, Ti
ICSAB	4400-061025MB01 Pace	500µg/mL	Al, Ca, Fe, K, Mg, Na, P, S,	10 mL	100 mL	50,000 ppb	Al, Ca, Fe, K, Mg, Na, S
		1,000µg/mL	C			100,000 ppb	C
		3,600µg/mL	Cl			360,000 ppb	Cl
		10µg/mL	Mo, Ti			1,000 ppb	Mo, Ti (see Next Section)
	T Int	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr,	8.0 mL		100 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Pd, Pt, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, U, Zr
		62.5 ppm	P (from two sources)			55,000 ppb	P
		0.625 ppm	Ag			50 ppb	Ag
		62.5 ppm	Si (from two sources)			5,000 ppb	Si
	Hg Int	1 ppm	Hg	0.50 mL		5 ppb	Hg

Table D:
WORKING STANDARDS continued

Standard	Stock or Intermediate Standard Used	Concentration	Elements	Amount Used	Final Volume (W/ Diluent)	Final Concentration	Elements
ICPMS Biota SPK ₁	PA-STD-1B	200 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn	5 mL	100 mL	10 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn
	PA-STD-3B	2,000 ppm	Al, Ca, Fe, K, Mg, Na	25 mL		500 ppm	Al, Ca, Fe, K, Mg, Na
	Single Stock Std.	10,000 ppm	K	15 mL		1,500 ppm	K (2000 ppm Total)
	Single Stock Std.	1,000 ppm	U	1 mL		10 ppm	U
	Single Stock Std.	1,000 ppm	Hg	0.025		0.25 ppm	Hg
	Conc. HNO ₃	69-70%		6.0 mL		6%	HNO ₃
ICPMS Biota SPK2 ₁	PA-STD-2B	1,000 ppm	Si	5.0 mL	100 mL	50 ppm	Si, P
		200 ppm	B, Mo, Sb, Sn, Ti, Zr			10 ppm	B, Mo, Sb, Sn, Ti, Zr
		100 ppm	Ag			5 ppm	Ag
	Conc. HNO ₃	69-70%		6.0 mL		6%	HNO ₃
ICP Water / Soil Spike ₂	PA-STD-1B	200 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Sr, Tl, V, Zn, P, Li	25 mL	200 mL	25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Sr, Tl, V, Zn, Li
	PA-STD-2B	1,000 ppm	Si	25 mL		125 ppm	Si, P
		200 ppm	B, Mo, Sb, Sn, Ti, Zr			25 ppm	B, Mo, Sb, Sn, Ti, Zr
		100 ppm	Ag			12.5 ppm	Ag
	PA-STD-3B	2,000 ppm	Al, Ca, Fe, K, Mg, Na	25 mL		250 ppm	Al, Ca, Fe, K, Mg, Na
ICPMS Water / Soil Spike ₂	Hg	1,000 ppm	Hg	0.025 mL	100 mL	250 ppm	Hg
	Pt	1,000 ppm	Pt	2.5mL		25000 ppm	Pt
	Pd	1,000 ppm	Pd	2.5mL		25000 ppm	Pd
	U	1,000 ppm	U	2.5 mL		25000 ppm	U
Internal Standards (ISTD)	PACE-WI-CAL-4A-Rev1	100 ppm	Bi, Ge, In, Sc, Tb, Y	2.0 mL	2000 mL	100 ppb	Bi, Ge, In, Sc, Tb, Y
	MSSC-100ppm	100 ppm	Sc	2.0 mL			
	Conc. HNO ₃	69-70%	NA	60 mL		3%	HNO ₃
	Gold	1,000ppm	Au	4.0mL		2,000 ppb	Au
Probe Rinse ₃	Gold	1,000ppm	Au	2.0	2500 mL	800 ppb	Au

Note: Equivalent substitutes may be used.

Note: Spikes may be altered as a result of client specific requirements.

1 = 1 mL added to LCS/LCSD and MS/MSD, (0.5 mL for Aqueous Digest), 0.2 mL to PS of 10 mL

2 = 1 mL added to LCS/LCSD and MS/MSD, (0.5 mL for Aqueous Digest), 0.2 mL to PS of 10 mL

3 = All Matrices contain 10% HNO₃ and 5% HCl

Table E: STOCK STANDARDS

Standard Type	Manufacturer	Part #	Conc.	Analytes	Used In
Calibration Stocks	SPEX CertiPrep	XFSMN-26-250A	100mg/L	As, Ba, Be, Cd, Co, Cr, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn, Cu	Calibration, CCVs, ICSAB
	SPEX CertiPrep	XFSMN-27-250A	500mg/L	Si	
			100mg/L	B, Mo, Sb, Sn, Ti, Zr	
			50mg/L	Ag	
			SPEX CertiPrep	XFSMN-28-250A	
	High-Purity Stds	100033-1	1,000µg/mL	Hg	
	Ultra Scientific	ICP-046	1,000µg/mL	Pt	
	Ultra Scientific	ICP-078	1,000µg/mL	Pd	
	Ricca	PSI10KW	10,000mg/L	Si	
	Fluka	19916-100mL	10,000mg/L	P	
Inorganic Ventures	AAU1-1	1,000mg/L	U		
ICV/Spike Stocks	Inorganic Ventures	PA-STD-1B	200mg/L	As, Ba, Be, Cd, Co, Cr, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn, Cu	ICV/Spike
	Inorganic Ventures	PA-STD-2B	1,000mg/L	Si	
			200mg/L	B, Mo, Sb, Sn, Ti, Zr	
			100mg/L	Ag	
			Inorganic Ventures	PA-STD-3B	
	Inorganic Ventures	CGHG1-1	1,000µg/mL	Hg	
	Inorganic Ventures	CGPT1-1	1,000µg/mL	Pt	
	Inorganic Ventures	CGPDN1-1	1,000µg/mL	Pd	
	High Purity Standards	100064-1	1,000µg/mL	U	
	Ultra Scientific	ICP-014	1,000mg/L	Si	
Ultra Scientific	ICO-015	1,000mg/L	P		
Internal Standard Stocks	Inorganic Ventures	PACEWI-CAL-4A-Rev1	100mg/L	Bi, Ge, In, Tb, Y	ISTD, Tune
	Inorganic Ventures	MSSC-100PPM	100mg/L	Sc	
Single Element Stocks	Ultra Scientific	ICP-112	10,000 mg/L	Mg	Tune / Int
	Ultra Scientific	ICP-056	1,000 mg/L	Ba	Tune
	Ultra Scientific	ICP-005	1,000 mg/L	B	Tune
	Ultra Scientific	ICP-004	1,000 mg/L	Be	Tune
	Ultra Scientific	ICP-058	1,000 mg/L	Ce	Tune
	Ultra Scientific	ICP-027	1,000 mg/L	Co	Tune
	Inorganic Ventures	CGLI10-5	10,000 mg/L	Li	Tune
	Ultra Scientific	ICP-028	1,000 mg/L	Ni	Tune
	Ultra Scientific	ICP-082	1,000 mg/L	Pb	Tune
	Inorganic Ventures	AAU1-1	1,000 mg/L	U	Tune / Cal
	High Purity Standards	100033-1	1,000 mg/L	Hg	Cal
	Ultra Scientific	ICP-119	10,000 mg/L	K	Spike
ICSA	Inorganic Ventures	6020ICS-0A	1,000µg/mL	Al, Ca, Fe, K, Mg, Na, P, S,	ICSA & ICSAB
			2,000µg/mL	C	
			10,000µg/mL	Cl	
			20µg/mL	Mo, Ti	

Note: Equivalent substitutes may be used.

Note: Single element standards may be used.

Table F:
INTERMEDIATE STANDARDS

Intermediate Type	Name	Stock Standard(s) Used (Part #)	Concentration	Elements	Amount Used	Acid Used	Final Volume (W/Nanopure H ₂ O)	Final Concentration
Tune Solution Intermediates	Mg Tune Int	ICP-112	10,000 ppm	Mg	0.1 mL	3 mL HNO ₃	100 mL	10 ppm
	Tune Int	Mg Tune Int	10 ppm	Mg	10 mL	2 mL HNO ₃	100 mL	1 ppm
		PACEWI-CAL-4A-REV1	100 ppm	Bi, Ge, In, Tb, Y	1.0 mL			
		ICP-056	1,000 ppm	Ba	0.1 mL			
		ICP-005	1,000 ppm	B	0.1 mL			
		ICP-004	1,000 ppm	Be	0.1 mL			
		ICP-058	1,000 ppm	Ce	0.1 mL			
		ICP-027	1,000 ppm	Co	0.1 mL			
		CGLI10-5	10,000 ppm	Li	0.01 mL			
		ICP-028	1,000 ppm	Ni	0.1 mL			
		ICP-082	1,000 ppm	Pb	0.1 mL			
		100064-1	1,000 ppm	U	0.1 mL			
		MSSC-100PPM	100 ppm	Sc	1.0 mL			
Calibration Intermediates	Hg Int	100033-1 CGAU1	1,000 ppm	Hg Au (preservative)	0.1 mL	3 mL HNO ₃	100 mL	1 ppm
	T Int	AAU1-1	1,000 ppm	U	0.25 mL	20 mL HNO ₃ 10 mL HCl	200 mL	1.25 ppm
		XFSMN-26-250A	100 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn	2.5mL			1.25 ppm
		XFSMN-27-250A	100 ppm Ag 50 ppm Si 500 ppm	Si, B, Mo, Sb, Sn, Ti, Zr, Ag	2.5 mL			1.25 ppm 2.5 ppm 6.25 ppm
		19916	10,000 ppm	P	1.225			61.25 ppm
		08729	10,000 ppm	Si	1.125			56.25 ppm
	M Int	XFSMN-28-250A	1,000 ppm	Al, Ca, Fe, K, Mg, Na	50 mL	3 mL HNO ₃	100 mL	500 ppm
ICV Intermediate	Hg ICV Int	CGHG1-1	1,000 ppm	Hg	1.0 mL	1 mL HNO ₃	100 mL	10 ppm
XCAL Int	Li XCAL Int	CGLI10	10,000 ppm	Li	0.1 mL	5 mL HNO ₃	50 mL	20 ppm

Table G:
REAGENTS

Reagent/Stock Std	Used In	Purity	Vendor	Order #
Hydrochloric Acid, (34-37% HCl)	Std. And Prep	Plasma Pure	SCP	250-038-155
Nitric Acid, (67-70% HNO ₃)	Std. And Prep	Plasma Pure	SCP	250-038-175
Nanopure or Milli-Q Water	all	18 Meg Ohms	On-Site	On-Site
Hydrogen Peroxide (30% H ₂ O ₂)	Prep	Trace Metals Grade	Fischer Scientific	H325-4

Reagents expire two years from receipt or on the manufacturer's expiration date, whichever is earliest.

Table H:
CALIBRATION LEVELS

Element	Cal 0 Blank	Calibration Standard 1 and RLVS as applicable	Calibration Standard 2 and RLVS as applicable	Calibration Standard 3	Calibration Standard 4	Calibration Standard 5
Aluminum	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Antimony	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Arsenic	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Barium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Beryllium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Boron	0 ppb		5 ppb	50 ppb	250 ppb	500 ppb
Cadmium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Calcium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Chromium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Cobalt	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Copper	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Iron	0 ppb	250 ppb	500ppb	2,500 ppb	12,500 ppb	25,000 ppb
Lithium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Lead	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Magnesium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Manganese	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Mercury	0 ppb	0.2 ppb	0.5 ppb	1.0 ppb	10 ppb	25 ppb
Molybdenum	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Nickel	0 ppb	1 ppb	5	50 ppb	250 ppb	500 ppb
Palladium	0 ppb	1 ppb	5	50 ppb	250 ppb	500 ppb
Phosphorus	0 ppb	50 ppb	250 ppb	2500 ppb	12500 ppb	25000 ppb
Platinum	0 ppb	1 ppb	5	50 ppb	250 ppb	500 ppb
Potassium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Selenium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Silicon	0 ppb	50 ppb	250 ppb	2500 ppb	12500 ppb	25000 ppb
Silver	0 ppb	0.5 ppb	2.5 ppb	25 ppb	125 ppb	250 ppb
Sodium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Strontium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Thallium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Tin	0 ppb	1 ppb Water Only	5 ppb	50 ppb	250 ppb	500 ppb
Titanium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Uranium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Vanadium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Zinc	0 ppb		5 ppb	50 ppb	250 ppb	500 ppb
Zirconium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Internal Standards ₁	50 ppb each	50 ppb each	50 ppb each	50 ppb each	50 ppb each	50 ppb each

Note: Calibration levels may change if noted. All solutions in 10% HNO₃ and 5% HCl.

1 = The Internal Standard Concentration is a result of a 2X Dilution via a T adapter in the delivery lines to the nebulizer.

Table I:
INTERNAL STANDARDS
(Multiple Listing Indicates Interpolation Between the Surrounding Internal Standards)

ISTD	Analytes
⁴⁵ Sc-CCT	Li, Be, B
⁴⁵ Sc-KED ⁷² Ge	Na – Zn
⁷² Ge ⁸⁹ Y	As – Sr
⁸⁹ Y ¹¹⁵ In	Mo – Cd
¹¹⁵ In ¹⁵⁹ Tb	Sn – Ba
¹⁵⁹ Tb ²⁰⁹ Bi	Hg – Pb
²⁰⁹ Bi	U

Table J:
EQUATIONS

Analyte	Recommended Elemental Interference Equations
⁴³ Ca ₂	-0.00135* ⁸⁸ Sr
⁴⁵ Sc-KED	-0.0399* ³¹ P -0.00025* ⁹⁰ Zr
⁴⁵ Sc-CCT	-0.06599* ³¹ P -0.00599* ²⁸ Si -0.00150* ⁹⁰ Zr
⁴⁷ Ti	-0.00030* ⁹⁰ Zr -0.00162* ³¹ P -0.00001* ⁹⁵ Mo
⁵¹ V	-2.0995* ⁵³ Cr
⁵³ Cr	-0.11400* ⁵² Cr
⁵² Cr	-0.00065* ³⁵ Cl
⁵⁵ Mn	-0.00125* ⁵⁴ Fe
⁵⁴ Fe	-0.03613* ⁵² Cr
⁵⁹ Co	-0.00063* ⁵⁴ Fe
⁶⁰ Ni	-0.00020* ⁴³ Ca -0.00059* ⁵⁹ Co -0.00007* ⁵⁴ Fe -0.00005* ¹¹⁸ Sn
⁶³ Cu	-0.00010* ⁶⁰ Ni
⁶⁶ Zn	-0.00010* ⁵⁴ Fe -0.00030* ¹³⁷ Ba
⁷² Ge	-0.00050* ⁵⁴ Fe
⁷⁵ As	-0.00001* ⁵⁹ Co
⁷⁸ Se	-0.03065* ⁸³ Kr
⁹⁰ Zr	-0.00154* ⁷³ Ge
⁹⁵ Mo	-0.00027* ⁹⁰ Zr
¹⁰⁷ Ag	-0.00055* ¹⁰⁵ Pd -0.00061* ⁹⁰ Zr

¹¹¹ Cd	-0.00020* ¹⁰⁵ Pd -0.00055* ⁹⁵ Mo -0.00003* ⁹⁰ Zr
¹¹⁵ In	-0.01416* ¹¹⁸ Sn
¹²¹ Sb	-0.00025* ¹¹⁸ Sn
¹³⁷ Ba	-0.00008* ¹²¹ Sb
²⁰¹ Hg	-0.00055 * ¹⁸⁴ W
²⁰⁸ Pb	1.00000 * ²⁰⁶ Pb + 1.00000 * ²⁰⁷ Pb
²⁰⁹ Bi	-0.00135 * ¹⁹⁵ Pt

1 – The equation may require periodic adjustment based on the KED add gas and tuning parameters. The mean value in counts per second (cps) for the calibration blank **MUST** be > 0 cps (ideally, all replicates should be > 0 cps). The normal operating range in cps is 0-1000cps for the calibration blank and must be inspected by the analyst.

2 – Both the ICSAB and the LCS sample are used in the evaluation of this equation. Ca is affected by doubly charged strontium and can vary from day to day plasma conditions. The ICSAB will not fully identify doubly charged conditions based on the ratio of Ca to Sr, whereas the LCS sample for waters and soils have sufficiently large Sr concentrations compared to Ca and will assist in identification of adjustment requirements for the Ca interference equation.

3 – Zirconium must be less than the linear dynamic range (LDR) of 10,000 µg/L on instrument.

Table K:
ACQUISITION/DWELL TIMES

Mass	Element	Resolution Mode	Dwell Time Per Mass (mSec)	Mass	Element	Resolution Mode	Dwell Time Per Mass (mSec)
7	Li	Standard	10	75	As	Standard	50
9	Be	Standard	10	78	Se	Standard	50
10	B	Standard	10	83	Kr	Standard	50
23	Na	High	5	89	Y	Standard	10
25	Mg	Standard	10	95	Mo	Standard	10
27	Al	Standard	10	105	Pd	Standard	10
28	Si	Standard	10	107	Ag	Standard	10
31	P	Standard	10	111	Cd	Standard	10
34	S	High	10	115	In	Standard	10
35	Cl	High	10	118	Sn	Standard	10
39	K	High	10	121	Sb	Standard	10
43	Ca	Standard	10	137	Ba	Standard	10
45	Sc-KED	Standard	10	159	Tb	Standard	10
45	Sc-CCT	High	10	184	W	Standard	10
47	Ti	Standard	10	195	Pt	Standard	10
51	V	Standard	10	201	Hg	Standard	10
52	Cr	Standard	10	205	Tl	Standard	10
54	Fe	Standard	10	206	Pb	Standard	10
55	Mn	Standard	10	207	Pb	Standard	10
59	Co	Standard	10	208	Pb	Standard	10
60	Ni	Standard	10	209	Bi	Standard	10
63	Cu	Standard	10	238	U	Standard	10
66	Zn	Standard	10	209	Bi	Standard	10
72	Ge	Standard	10	238	U	Standard	10

Table L:
INSTRUMENT QC LIMITS

Element	ICV (ppb)	CCV (ppb)	ICV/CCV Limits	ICSA (ppb)	ICSAB (ppb)	ICSAB Limits	RLVS (ppb)	RLVS Limit
Aluminum	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Antimony	110	100	90%-110%		100	80%-120%	1	70-130%
Arsenic	110	100	90%-110%		100	80%-120%	1	70-130%
Barium	110	100	90%-110%		100	80%-120%	1	70-130%
Beryllium	110	100	90%-110%		100	80%-120%	1	70-130%
Boron	110	100	90%-110%		100	80%-120%	5	70-130%
Cadmium	110	100	90%-110%		100	80%-120%	1	70-130%
Calcium	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Chromium	110	100	90%-110%		100	80%-120%	1	70-130%
Cobalt	110	100	90%-110%		100	80%-120%	1	70-130%
Copper	110	100	90%-110%		100	80%-120%	1	70-130%
Iron	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Lithium	110	100	90%-110%		100	80%-120%	1	70-130%
Lead	110	100	90%-110%		100	80%-120%	1	70-130%
Magnesium	110	100	90%-110%	50,000	50,000	80%-120%	250	70-130%
Manganese	11000	10000	90%-110%		100	80%-120%	1	70-130%
Mercury	4	5	90%-110%		5	80%-120%	0.2	70-130%
Molybdenum	110	100	90%-110%	1,000	1,100	80%-120%	1	70-130%
Nickel	110	100	90%-110%		100	80%-120%	1	70-130%
Palladium	110	100	90%-110%		100	80%-120%	1	70-130%
Phosphorus	5500	5000	90%-110%	50000	55000	80%-120%	50 Water 250 Soil/Biota	70-130%
Platinum	110	100	90%-110%		100	80%-120%	1	70-130%
Potassium	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Selenium	110	100	90%-110%		100	80%-120%	1	70-130%
Silicon	5500	5000	90%-110%		5000	80%-120%	50 Water Only	70-130%
Silver	55	50	90%-110%		50	80%-120%	0.5	70-130%
Sodium	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Strontium	110	100	90%-110%		100	80%-120%	1	70-130%
Thallium	110	100	90%-110%		100	80%-120%	1	70-130%
Tin	110	100	90%-110%		100	80%-120%	1 Water 5 Soil/Biota	70-130%
Titanium	110	100	90%-110%	1,000	1,100	80%-120%	1	70-130%
Uranium	110	100	90%-110%		100	80%-120%	1	70-130%
Vanadium	110	100	90%-110%		100	80%-120%	1	70-130%
Zinc	110	100	90%-110%		100	80%-120%	5	70-130%
Zirconium	110	100	90%-110%		100	80%-120%	1	70-130%

Table M:
AQUEOUS BATCH QC
ON INSTRUMENT
CONCENTRATIONS AND LIMITS

Element	LCS/ LCSD (ppb)	LCS/LCSD Limits (%)	MS/MSD Amount (ppb)	MS/MSD Limits (%)	PS Amount (ppb)	PS Limits (%)
Aluminum	5,000	80-120	5,000	75-125	5,000	80-120
Antimony	500	80-120	500	75-125	500	80-120
Arsenic	500	80-120	500	75-125	500	80-120
Barium	500	80-120	500	75-125	500	80-120
Beryllium	500	80-120	500	75-125	500	80-120
Boron	500	80-120	500	75-125	500	80-120
Cadmium	500	80-120	500	75-125	500	80-120
Calcium	5,000	80-120	5,000	75-125	5,000	80-120
Chromium	500	80-120	500	75-125	500	80-120
Cobalt	500	80-120	500	75-125	500	80-120
Copper	500	80-120	500	75-125	500	80-120
Iron	5,000	80-120	5,000	75-125	5,000	80-120
Lithium	500	80-120	500	75-125	500	80-120
Lead	500	80-120	500	75-125	500	80-120
Magnesium	5,000	80-120	5,000	75-125	5,000	80-120
Manganese	500	80-120	500	75-125	500	80-120
Mercury	5	80-120	5	75-125	5	80-120
Molybdenum	500	80-120	500	75-125	500	80-120
Nickel	500	80-120	500	75-125	500	80-120
Palladium	500	80-120	500	75-125	500	80-120
Phosphorus	500	80-120	500	75-125	500	80-120
Platinum	500	80-120	500	75-125	500	80-120
Potassium	5,000	80-120	5,000	75-125	5,000	80-120
Selenium	500	80-120	500	75-125	500	80-120
Silicon	2500	80-120	2500	75-125	2500	80-120
Silver	250	80-120	250	75-125	250	80-120
Sodium	5,000	80-120	5,000	75-125	5,000	80-120
Strontium	500	80-120	500	75-125	500	80-120
Thallium	500	80-120	500	75-125	500	80-120
Tin	500	80-120	500	75-125	500	80-120
Titanium	500	80-120	500	75-125	500	80-120
Uranium	500	80-120	500	75-125	500	80-120
Vanadium	500	80-120	500	75-125	500	80-120
Zinc	500	80-120	500	75-125	500	80-120
Zirconium	500	80-120	500	75-125	500	80-120

Please see the QA manager for the most current LODs.

Table N:
SOLIDS BATCH QC
ON INSTRUMENT
CONCENTRATIONS AND LIMITS

Element	LCS/ LCSD (ppb)	LCS/LCSD Limits (%)	MS/MSD Amount (ppb)	MS/MSD Limits (%)	PS Amount (ppb)	PS Limits (%)
Aluminum	5000	80-120	5000	75-125	5,000	80-120
Antimony	500	80-120	500	75-125	500	80-120
Arsenic	500	80-120	500	75-125	500	80-120
Barium	500	80-120	500	75-125	500	80-120
Beryllium	500	80-120	500	75-125	500	80-120
Boron	500	80-120	500	75-125	500	80-120
Cadmium	500	80-120	500	75-125	500	80-120
Calcium	5000	80-120	5000	75-125	5,000	80-120
Chromium	500	80-120	500	75-125	500	80-120
Cobalt	500	80-120	500	75-125	500	80-120
Copper	500	80-120	500	75-125	500	80-120
Iron	5000	80-120	5000	75-125	5,000	80-120
Lithium	500	80-120	500	75-125	500	80-120
Lead	500	80-120	500	75-125	500	80-120
Magnesium	5000	80-120	5000	75-125	5,000	80-120
Manganese	500	80-120	500	75-125	500	80-120
Mercury	5	80-120	5	75-125	5	80-120
Molybdenum	500	80-120	500	75-125	500	80-120
Nickel	500	80-120	500	75-125	500	80-120
Phosphorus	500	80-120	500	75-125	500	80-120
Potassium	5000	80-120	5000	75-125	5,000	80-120
Selenium	500	80-120	500	75-125	500	80-120
Silicon	2500	80-120	2500	75-125	2,500	80-120
Silver	250	80-120	250	75-125	250	80-120
Sodium	5000	80-120	5000	75-125	5,000	80-120
Strontium	500	80-120	500	75-125	500	80-120
Thallium	500	80-120	500	75-125	500	80-120
Tin	500	80-120	500	75-125	500	80-120
Titanium	500	80-120	500	75-125	500	80-120
Uranium	500	80-120	500	75-125	500	80-120
Vanadium	500	80-120	500	75-125	500	80-120
Zinc	500	80-120	500	75-125	500	80-120
Zirconium	500	80-120	500	75-125	500	80-120

Please see the QA manager for the most current LODs.

Table O:
BIOTA BATCH QC ON INSTRUMENT
CONCENTRATIONS AND LIMITS

Element	LCS/ LCSD (ppb)	LCS/LCSD Limits (%)	MS/MSD Amount (ppb)	MS/MSD Limits (%)	PS Amount (ppb)	PS Limits (%)
Aluminum	10,000	80-120	10,000	75-125	10,000	80-120
Antimony	200	80-120	200	75-125	200	80-120
Arsenic	200	80-120	200	75-125	200	80-120
Barium	200	80-120	200	75-125	200	80-120
Beryllium	200	80-120	200	75-125	200	80-120
Boron	200	80-120	200	75-125	200	80-120
Cadmium	200	80-120	200	75-125	200	80-120
Calcium	10,000	80-120	10,000	75-125	10,000	80-120
Chromium	200	80-120	200	75-125	200	80-120
Cobalt	200	80-120	200	75-125	200	80-120
Copper	200	80-120	200	75-125	200	80-120
Iron	10,000	80-120	10,000	75-125	10,000	80-120
Lithium	500	80-120	500	75-125	500	80-120
Lead	200	80-120	200	75-125	200	80-120
Magnesium	10,000	80-120	10,000	75-125	10,000	80-120
Manganese	200	80-120	200	75-125	200	80-120
Mercury	5	80-120	5	75-125	5	80-120
Molybdenum	200	80-120	200	75-125	200	80-120
Nickel	200	80-120	200	75-125	200	80-120
Phosphorus	200	80-120	200	75-125	20,000	80-120
Potassium	40,000	80-120	40,000	75-125	40,000	80-120
Selenium	200	80-120	200	75-125	200	80-120
Silicon	1000	80-120	1000	75-125	1,000	80-120
Silver	100	80-120	100	75-125	100	80-120
Sodium	10,000	80-120	10,000	75-125	10,000	80-120
Strontium	200	80-120	200	75-125	200	80-120
Thallium	200	80-120	200	75-125	200	80-120
Tin	200	80-120	200	75-125	200	80-120
Titanium	200	80-120	200	75-125	200	80-120
Uranium	200	80-120	200	75-125	200	80-120
Vanadium	200	80-120	200	75-125	200	80-120
Zinc	200	80-120	200	75-125	200	80-120
Zirconium	200	80-120	200	75-125	200	80-120

Note: Biota spikes are fortified for K as a result of its natural presence in the Chicken blank.

Please see the QA manager for the most current LODs.

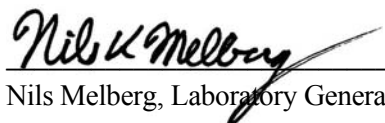
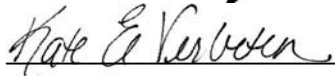
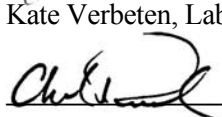
STANDARD OPERATING PROCEDURE

The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy – CETAC M-7500

Reference Methods: EPA SW-846 Methods 7470A and 7471B, EPA 245.1

SOP NUMBER:	S-GB-M-017-REV.04
EFFECTIVE DATE:	Date of Final Signature
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APPROVAL

 _____ Nils Melberg, Laboratory General Manager	_____ 06/13/16 Date
 _____ Kate Verbeten, Laboratory Quality Manager	_____ 6/13/2016 Date
 _____ Chad Rusch, Department Manager	_____ 06/13/2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

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_____ Signature	_____ Title	_____ Date
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1. Purpose/Identification of Method

- 1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the determination of mercury in water, soil/solid samples, and biological tissue analyzed by the Cetac M-7500 instrument by EPA Methods SW846 7470A, SW846 7471B and EPA 245.1.

2. Summary of Method

- 2.1 Cold vapor atomic absorption utilizes the volatile property of elemental mercury at the 253.7 nm wavelength. To release mercury from organic complexes, the sample is digested with oxidizing reagents and acids in a hot block. After digestion, the oxidizing reagents are neutralized. Stannous chloride is added to reduce ionic mercury to the ground state. The Flow Injection Analysis System sweeps the volatile elemental mercury out of the sample and into the cell of an atomic absorption spectrophotometer. The absorbance signal is proportional to the amount of mercury in the sample.

3. Scope and Application

- 3.1 This method is applicable to water samples (including surface water and domestic and industrial wastewaters), solid samples (including soils, sediments, sludge, and solid wastes), wipes, TCLP, SPLP and ASTM leachates, and biological tissue samples.
- 3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by atomic absorption spectrometry. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

- 4.1 7470A and EPA 245.1 apply to aqueous samples including surface water, dissolved water, domestic and industrial wastewaters, TCLP, SPLP, and ASTM leachates.
- 4.2 7471B applies to soil and solid samples.
- 4.3 7471M is a modified 7471B method that applies to biological tissue samples

5. Limits of Detection and Quantitation

- 5.1 All current LODs and LOQs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

- 6.1 Samples can contain diverse matrix types, each of which may present analytical challenges. Spiked samples and Laboratory Control Samples are important for determining digestion efficiency.
- 6.2 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide, as NaS, do not interfere with recovery of added inorganic Mercury from D.I. water.

- 6.3 Copper has also been reported to interfere; however, copper concentrations as high as 10mg/L had no effect on recovery of Mercury from spiked samples.
- 6.4 Samples high in chlorides require up to 7.5 mL of additional potassium permanganate. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the Mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent. Samples may be diluted to decrease the chlorides. Alternatively, the sample may be allowed to stand for at least an hour under a hood (without active purging) to remove the chlorine.
- 6.5 Certain volatile organic materials that absorb at this wavelength may also interfere. A preliminary run without reagents would determine if this type of interference is present.

7. Sample Collection, Preservation, Shipment and Storage

7.1 Biota Samples

- 7.1.1 Biota samples can be collected in clean plastic or glass containers or plastic zip-top bags.
- 7.1.2 Biota samples are kept frozen at $\leq -10^{\circ}\text{C}$ until time of preparation to preserve integrity. Hold time is 28 days from removal from freezer.

7.2 Soil Samples

- 7.2.1 Soil and waste samples should be collected in clean wide-mouth glass containers to facilitate obtaining representative aliquots for measurement.
- 7.2.2 Samples should be stored at $\leq 6^{\circ}\text{C}$ and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.3 Wipe Samples

- 7.3.1 Wipe should be collected in clean wide-mouth glass containers or a digestion vessel.
- 7.3.2 Samples should be stored at $\leq 6^{\circ}\text{C}$ and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.4 Aqueous Samples

- 7.4.1 Water samples may be collected in plastic or glass containers and must be preserved with HNO_3 to pH < 2 and stored at room temperature, acid not to exceed 2% of the estimated sample volume.

NOTE: Aqueous samples that react violently to the addition of acid may be collected without chemical preservation with proper variances approved by the regulatory authority. The responsibility of requesting this variance lies with the sample collector.

7.4.2 NOTE: Samples may be preserved in the lab. The samples may not be processed until 24 hours after preservation with a pH test of <2 . Appropriate qualifier added if $\text{pH} > 2$ after preservation. Dissolved samples should be field filtered through a 0.45 micron filter prior to preservation. Samples can be filtered in the lab, but must be received unpreserved and will be qualified as not being preserved properly. A filter blank must be created when lab filtering and analyzed using method blank acceptance criterion to demonstrate the process is not affecting the data quality.

7.4.3 Samples must be analyzed within 28 days of collection.

7.5 TCLP, SPLP, and ASTM Samples

7.5.1 Leach extracts should be collected in plastic or glass containers and must be refrigerated at $\leq 6^{\circ}\text{C}$. Samples are preserved at time of digestion because leach volumes vary. The MS and MSD are not to be preserved until after they are spiked.

7.5.2 NOTE: TCLP Samples are not required to sit for 24 hours prior to digestion after the addition of nitric acid. Samples must be extracted within 28 days from field collection to leach extraction. Samples must be digested and analyzed within 28 days of the leach extraction. Total elapsed hold time is 56 days.

8. Definitions

8.1 Definitions of terms found in this SOP can also be found in the Pace Quality Manual. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP or the Pace Analytical Services' Quality Manual Glossary, Section 10.0.

8.2 Biota Control Blank (Matrix Blank) – A sample of a matrix that is used for the control spike. The biota control blank will either be catfish, tilapia, chicken, or other tissue whichever is available at the time of analysis. The biota control blank should be “farm-raised” to minimize background mercury levels. The concentration in the biota control blank will be subtracted from the concentration of the laboratory control spike when the biota control blank concentration is greater than/equal to the MDL. This is done because the biota control blank is known to have some contamination. This SOP will reference biota control blank for ease. Note: For plant material analysis, alfalfa can be used as the biota control blank matrix.

8.3 Reagent Grade – Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

- 8.4 Standardized Reference Material (SRM) – A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. A SRM is analyzed with each analytical batch of biota samples.
- 8.5 Pace Reporting Limit (PRL) – The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

9. Equipment and Supplies

- 9.1 Hot Block – Operated at $95 \pm 3^{\circ}\text{C}$. Heating block should be Environmental Express “Hot Block”, or equivalent.
- 9.2 Digestion Vials – Clean 15- mL screw cap vials, Environmental Express (SC415), or equivalent.
- 9.3 Digestion Vials- Clean 68-mL screw cap vials, Environmental Express (SC475), or equivalent.
- 9.4 Volumetric Flasks – Assorted, Class A
- 9.5 Graduated Cylinder – 50-mL
- 9.6 Mechanical Pipettes – Assorted adjustable air-displacement pipettes with disposable tips (Eppendorf or equivalent).
- 9.7 pH Strips
- 9.8 CETAC M-7500 with auto-sampler
- 9.9 Inert Sparging and Carrier Gas – Argon
- 9.10 Autosampler vials
- 9.11 Autosampler Pump Tubing
- 9.12 Specimen Cups
- 9.13 DORM-4 or equivalent
- 9.14 1570a (Trace Elements in Spinach) or equivalent
- 9.15 Analytical Balance – Capable of weighing to 0.001g

10. Reagents and Standards

- 10.1 Trace Metals Water – Nano-Pure Water
- 10.2 Nitric Acid (HNO_3) – 9598-34 J.T. Baker, or equivalent. The reagent is assigned a two year expiration date, not to exceed the manufacturer's expiration date.
- 10.3 Hydrochloric Acid (HCl) – 9530 -33 J.T. Baker, or equivalent. The reagent is assigned a two year expiration date, not to exceed the manufacturer's expiration date.
- 10.4 Sulfuric Acid (H_2SO_4) – 9681-33 J.T. Baker, or equivalent. The reagent is assigned a two year expiration date, not to exceed the manufacturer's expiration date.
- 10.5 KmnO_4 neat – P279-500 Fisher Scientific. The reagent is assigned a five year expiration date, not to exceed the manufacturer's expiration date.
- 10.6 $\text{K}_2\text{S}_2\text{O}_8$ neat – 3238-01 J.T. Baker. The reagent is assigned a five year expiration date, not to exceed the manufacturer's expiration date.
- 10.7 $\text{NH}_2\text{OH}\cdot\text{HCl}$ neat – H330-500 Fisher Scientific. The reagent is assigned a five year expiration date, not to exceed the manufacturer's expiration date.
- 10.8 SnCl_2 neat – T142-500 Fisher Scientific. The reagent is assigned a five year expiration date, not to exceed the manufacturer's expiration date.
- 10.9 Potassium Permanganate (KmnO_4) solution (5%) – Dissolve 50.0 g of KmnO_4 into ~800 mL of trace metals water. Dilute to 1000 mL. The reagent is assigned a one month expiration date.
- 10.10 Potassium Persulfate ($\text{K}_2\text{S}_2\text{O}_8$) solution (5%) – Dissolve 50.0 g of $\text{K}_2\text{S}_2\text{O}_8$ into ~800 mL of trace metals water. Dilute to 1000 mL. Warm to dissolve. The reagent is assigned a one month expiration date.
- 10.11 Hydroxylamine Hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) Solution (12%) – Dissolve 120 g NaCl and 120g $\text{NH}_2\text{OH}\cdot\text{HCl}$ in ~800 mL of trace metals water. Dilute to 1000 mL. The reagent is assigned a one month expiration date.
- 10.12 Stannous Chloride Solution (SnCl_2) – Dissolve 100.0 g of SnCl in ~ 800 mL of trace metals water. Add 70 mL of HCl . Dilute to 1000 mL. The reagent is assigned a one week expiration date. Discard if oxidized or precipitate forms.
- 10.13 Acid Rinse Solution- Add 125 mL of HCl and 125 mL concentrated Nitric acid in ~1000 mL of trace metals water dilute to 2,500 mL. The reagent is assigned a six month expiration date.
- 10.14 Diluent – Each method diluent needed is digested weekly and assigned a one week expiration date. Diluent is made of digested blanks, digested according to method and SOP procedures.

- 10.15 Analytical Standards – Standard solutions are required for calibration, calibration checks, and sample spiking solutions. The following describes the contents of each type of solution. The commercial sources for stock solutions, recipes for preparing dilutions and working standards, and concentrations in standard solutions are presented in Table 1, Table 2, and Table 3.

Table 1: Standard Definitions

Standard 7470A/7471B	Standard 245.1	Description	Comments
Initial Calibration Standard(s)	Initial Calibration Standard(s)	Standards prepared from single and/or multi-element standard(s) at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range for single point calibration.	Working standards are made daily.
Initial Calibration Verification Standard (ICV)	Quality Control Sample (QCS) & 1 st IPC	Standard prepared near the midpoint of the calibration range and is used to verify the accuracy of the calibration and instrument performance.	Must be prepared from a source independent of CCV and Calibration Solutions.
Continuing Calibration Verification Standard (CCV)	Instrument Performance Check Solution (IPC)	Standards prepared from single and/or multi-element standards at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range. This standard verifies the accuracy of the calibration curve.	May be prepared from the same source as the calibrations standards
Reporting Limit Verification Standard (RLVS)	NA	Must be performed after ICV/ICB and prior to samples. For 7471B it is also required at the end of the analytical sequence. For 7471B the recovery must be between 70 and 130% to report sample data. All other methods must be between 60-140%.	NELAC requirement, a client specific requirement for certain QAPPs
Single Element Standards	Single Element Standards	Stock standards purchased from vendors containing one element. Used for checking IECs and may be used for checking linear ranges.	Must be 99.99% pure.
Spiking Standard	Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	Prepared from a source independent of CCV and Calibration Solutions.
Method Blank (MB)	Laboratory Reagent Blank (LRB)	This blank must contain all the reagents, in the same volumes as used for samples and must be carried through the complete processing of samples with the samples.	
Laboratory Control Sample (LCS)	Laboratory Fortified Blank (LFB)	Lab water spiked with the reagents of interest at a known concentration. It must contain all the reagents, in the same volumes as used for samples and must be carried through the complete processing of samples with the samples.	Spiked from a source independent of CCV and Calibration Solutions.
Matrix Spike (MS), Matrix Spike Duplicate (MSD)	Laboratory Fortified Sample Matrix (LFM)	Aliquots of environmental sample spiked with known concentrations of target analytes.	Spiked from a source independent of CCV and Calibration Solutions.

Note: This SOP will utilize the terms for standards listed in the 7470A/7471B column.

Table 2: Stock Standards

Standard	Concentration	Vendor	Catalog #
Hg Calibration Stock	1000µg/mL	High-Purity Standards	CGHG1-1
ICV Calibration Stock	1000µg/mL	Inorganic Ventures, Inc.	100033-1

Standards are used until they expire based on the manufacturer's expiration date.

Table 3: Intermediate and Calibration Standards 7471B and 7471M for Tissue

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO ₃ used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate (100 ug/L CVAA Hg Spk)	Hg ICV Stock	1000 µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 0	-	-	-	-	50 mL	0.0 ppb
Hg Cal. 1 and PRLs	Hg Cal. Intermediate	100 ppb	100 µL	-	50 mL	0.2 ppb
Hg Cal. 2	Hg Cal. Intermediate	100 ppb	500 µL	-	50 mL	1.0 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	1250 µL	-	50 mL	2.5 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	2500 µL	-	50 mL	5.0 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	5000 µL	-	50 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	2000 µL	-	50 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	2500 µL	-	50 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	50 mL	0.0 ppb

All intermediate dilution solutions have a 1 month expiration (This standard expiration time is based on Section 7.9 of EPA 1631 Revision E, August 2002). Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

Table 4: Intermediate and Calibration Standards 7470A and 245.1

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO ₃ used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate (100 ug/L CVAA Hg Spk)	Hg ICV Stock	1000 µg/mL	20 µL	6.0 mL	200 mL	100 ppb
1000 ug/L CVAA Hg Spk	Hg ICV Stock	1000 µg/mL	100 µL	3.0 mL	100 mL	1000 ppb
Hg Cal. 0	-	-	-	-	10 mL	0.0 ppb
Hg Cal. 1 and PRLS	Hg Cal. Intermediate	100 ppb	20 µL	-	10 mL	0.2 ppb
Hg Cal. 2	Hg Cal. Intermediate	100 ppb	100 µL	-	10 mL	1.0 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	250 µL	-	10 mL	2.5 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	500 µL	-	10 mL	5.0 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	1000 µL	-	10 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	400 µL	-	10 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	500 µL	-	10 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	10 mL	0.0 ppb

All intermediate dilution solutions have a 1 month expiration (This standard expiration time is based on Section 7.9 of EPA 1631 Revision E, August 2002). Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

11. Calibration and Standardization

- 11.1 A digested calibration curve is made up every time there are samples prepared. The curve is associated to the sample batch(es) it is prepped with. Calibration requires analysis of a calibration blank and at least five levels of calibration solutions. The lowest calibration concentration is the pace reporting limit.
- 11.2 The resultant correlation coefficient must be greater than 0.995. If this does not occur, then the instrument must be recalibrated and or the calibration curve and the associated samples must be re-prepped.
- 11.3 The calibration curve must pass an Initial Calibration Verification (ICV) that is analyzed after the calibration standards and before any samples. The ICV concentration is near the midpoint of the calibration curve and is made from a source other than the one used to make the calibration standards.
- 11.4 Pace Reporting Limit Standard (PRL or CRDL Std.) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration (typically after the ICB) with recovery 70-130% of true value for 7471B. All other methods must recover within 60-140%. If outside the limits, reanalyze once. If still outside the limits, recalibrate. When analyzing by 7471B, CRDLs must, at a minimum, bracket all samples.
- 11.5 Every ten samples or less must be bracketed by Continuing Calibration Verifications (CCV). The CCV concentration is near the midpoint of the calibration curve.
- 11.6 For 7470A, 7471M and 7471B the ICV and CCV limits are $\pm 10\%$ of their expected values. For 245.1 the ICV acceptance limits are $\pm 5\%$ and the CCV limits are $\pm 10\%$ of their expected values. If outside the limits, reanalyze once. If still outside the limits, recalibrate.
- 11.7 An acceptable Initial Calibration Blank (ICB) must be analyzed after the ICV.
- 11.8 Continuing Calibration Blanks (CCBs) are analyzed after the CCVs.
- 11.9 The control limit for the ICB and CCB is the absolute value, less than the Pace Reporting Limit. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12. Procedure

12.1 Sample Preparation

12.1.1 Water Samples – 7470A and 245.1

- 12.1.1.1 Verify preservation in the samples by checking their pH with a test strip (completed upon receipt in Sample Receiving Department).
- 12.1.1.2 Prior to analysis, the samples and the calibration curve must be digested.

- 12.1.1.3 Turn on the hot block and set the temperature to maintain a sample temperature of $95\pm 3^{\circ}\text{C}$. Before placing samples in the hot block, verify that the temperature is correct and adjust as required. Record the temperature in the sample logbook. Verify that the hood is functioning.
- 12.1.1.4 Use the 15 mL digestion tubes.
- 12.1.1.5 Add 10 mL of trace metals water minus the volume of standard to be added and add the appropriate amounts (as shown in Table 4) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.
- 12.1.1.6 Add 10 mL of trace metals water to a digestion vial for the Method Blank.
- 12.1.1.7 Add 10.0 mL of trace metals water and 0.05 mL of 1000 ug/L CVAA Hg Spk to two digestion vials for the Laboratory Control Spike and Laboratory Control Spike Duplicate. Only prep LCSD if requested by client or there is not enough sample to run an MS/MSD.
- 12.1.1.8 Add 10 mL of each well-mixed sample to digestion vials.
- 12.1.1.9 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random or assigned by the client, at a frequency of 5% for 7470A and 7471B methods and 10% for 245.1. An additional 10 mL of this sample, along with 0.05 mL of 1000 ug/L CVAA Hg Spk, is added to two digestion vials.
- NOTE: TCLP, SPLP, and ASTM extracts must be spiked prior to nitric acid preservation. One MS must be prepared per sample matrix (ie: soil and wood chips would each require a separate matrix spike).
- 12.1.1.10 To each digestion vial add 0.25 mL of concentrated nitric acid. Swirl the digestion vials gently.
- 12.1.1.11 To each digestion vial add 0.5 mL of concentrated sulfuric acid. Swirl the digestion vials gently.
- 12.1.1.12 Add 1.5 mL of 5% KmnO_4 , swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, use a smaller amount of a fresh aliquot of sample.
- 12.1.1.13 Add 0.8 mL of 5% Persulfate solution. Place lid or cap on tube so as to allow pressure to vent but minimizing evaporation. Swirl the digestion vials gently.
- 12.1.1.14 Digest samples for 2 hours at $95\pm 3^{\circ}\text{C}$.
- 12.1.1.15 Remove samples and let cool to room temperature.

12.1.1.16 Add 0.5 mL of 12% hydroxylamine hydrochloride solution. Shake the digestion vials gently to clear the KmnO_4 .

12.1.1.17 Sample volumes are consistent and said to be 10 mL. Samples are now ready for analysis.

12.1.2 Soil/Solid/Wipe Samples – 7471B

12.1.2.1 Prior to analysis the samples and calibration curve must be digested.

12.1.2.2 Turn on the hot block and set the temperature to maintain a sample temperature of $95 \pm 3^\circ\text{C}$. Before placing samples in the hot block to digest, verify that the temperature is correct and adjust as required. Record the temperature in the electronic prep log. Verify that the hood is functioning.

12.1.2.3 Use the 50 mL digestion tubes for soils, solid, and wipe samples.

12.1.2.4 Add 2.5 mL of Nano-Pure water and add the appropriate amounts (as shown in Table 3) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

12.1.2.5 Prepare one Method Blank by adding glass beads (or equivalent) to a digestion vial.

12.1.2.6 Prepare the Laboratory Control Spike (LCS) for soils, wipes, and wastes by adding glass beads (or equivalent) to a digestion vial. Then, add 2.5 mL of Hg ICV Intermediate (100 ug/L CVAA Hg Spk) to the digestion vial. Only prep a Laboratory Control Spike Duplicate (LCSD) if requested by client or there is not enough sample to run an MS/MSD. Prepare the LCSD in the same fashion as the LCS.

12.1.2.7 Weigh 0.3 to 0.34 g of homogenized sample into a labeled digestion vial. Record the weight to the nearest 0.01 g in the digestion prep log. For samples with high liquid content, a larger sample size may be used, as long as the digestion is complete. For wipes, put the WHOLE wipe in the digestion vial.

12.1.2.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random. Weigh 2 additional 0.3g aliquots into digestion vials. Add 2.5 mL of Hg ICV Intermediate (100 ug/L CVAA Hg Spk) to each vial.

12.1.2.9 Add 2.5 mL of Nano-pure water to all QC and samples. Swirl digestion vials to gently mix.

12.1.2.10 To each digestion vial add 0.7 mL of concentrated nitric acid. Swirl digestion vials to gently mix.

- 12.1.2.11 To each digestion vial add 2.1 mL of concentrated hydrochloric acid. Swirl digestion vials to gently mix.
 - 12.1.2.12 Heat for 2 minutes on a block set to reach $95\pm 3^{\circ}\text{C}$ and then allow the samples to cool.
 - 12.1.2.13 Add about 25 mL of Nano-pure water to all QC and samples. Swirl digestion vials to gently mix.
 - 12.1.2.14 Slowly add 7.5 mL of 5% KmnO_4 to all samples and batch QC. Samples may react violently if added too quickly.
 - 12.1.2.15 Place cap on tube so as to allow pressure to vent but minimizing evaporation, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, use a smaller amount of a fresh aliquot of sample.
 - 12.1.2.16 Digest samples for 30 minutes at $95\pm 3^{\circ}\text{C}$.
 - 12.1.2.17 Remove samples and let cool to room temperature.
 - 12.1.2.18 Bring to a final volume of 50mL with nanopure.
 - 12.1.2.19 Add 3.0 mL of 12% hydroxylamine hydrochloride solution. Cap the digestion vials and gently shake to clear KmnO_4 . Samples are consistent and said to have a final volume of 50 mL. Samples are now ready for analysis.
- 12.1.3 Biota Samples – 7471M
- 12.1.3.1 50 mL digestion vials are used for Biota samples.
 - 12.1.3.2 Prior to analysis the samples and calibration curve must be digested.
 - 12.1.3.3 Turn on the hot block and set the temperature to maintain a sample temperature of $95\pm 3^{\circ}\text{C}$. Before placing samples in the hot block to digest, verify that the water temperature is correct and adjust as required. Record the temperature in the electronic prep log. Verify that the hood is functioning.
 - 12.1.3.4 Add 2.5 mL of Nano-Pure water and add the appropriate amounts (as shown in Table 3) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.
 - 12.1.3.5 Prepare one Method Blank. For biota analysis, leave the Method Blank empty. A Biota Control Blank is also prepared by weighing 0.6 g of homogenized biota control blank tissue into a digestion vial.

- 12.1.3.6 Prepare the LCS for biota samples by weighing 0.6 g of homogenized biota control blank into the digestion vial. Add 2.5 mL of Hg ICV Intermediate (100 ug/L CVAA Hg Spk) the vial. Only prep a Laboratory Control Spike Duplicate (LCSD) if requested by client or there is not enough sample to run a MS/MSD. Prepare the LCSD in the same fashion as the LCS.
- 12.1.3.7 For biota samples, weigh approximately 0.05 g of Standard Reference Material into a labeled digestion vial. If biota samples are plants, use approximately 0.6 g of SRM 1570a. The analytical balance must read to at least 4 places past the decimal.
- 12.1.3.8 Weigh 0.6 g of homogenized sample into a labeled digestion vial. Record the weight to the nearest 0.01 g in the digestion prep log.
- 12.1.3.9 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random. Weigh 2 additional 0.6g aliquots into digestion vials. Add 2.5 mL of Hg ICV Intermediate (100 ug/L CVAA Hg Spk) to each vial.
- 12.1.3.10 To each digestion vial add 0.7 mL of concentrated nitric acid. Swirl digestion vials to gently mix.
- 12.1.3.11 To each digestion vial add 2.1 mL of concentrated hydrochloric acid. Swirl digestion vials to gently mix.
- 12.1.3.12 Heat for 2 minutes on a block set to reach $95\pm3^{\circ}\text{C}$ and then allow the samples to cool.
- 12.1.3.13 Add about 25 mL of Nano-pure water to all QC and samples. Swirl digestion vials to gently mix.
- 12.1.3.14 Slowly add 7.5 mL of 5% KmnO_4 to all samples and batch QC. Samples may react violently if added too quickly.
- 12.1.3.15 Place cap on tube so as to allow pressure to vent but minimizing evaporation, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, use a smaller amount of a fresh aliquot of sample
- 12.1.3.16 Digest samples for 30 minutes at $95\pm3^{\circ}\text{C}$.
- 12.1.3.17 Remove samples and let cool to room temperature.
- 12.1.3.18 Bring to a final volume of 50 mL with nanopure.
- 12.1.3.19 Add 3.0 mL of 12% hydroxylamine hydrochloride solution. Cap digestion vials and gently shake to clear KmnO_4 . Samples are consistent and said to have a final volume of 50 mL. Samples are now ready for analysis.

12.2 Basic System Operation – Analytical: This portion of the SOP is designed to allow the user to set up and run a method, print a sample report, then shut down using the more basic software functions. For a more detailed explanation of the many other options, the user should refer to the Reference Manual.

12.2.1 Verify the carrier argon gas supply is on.

12.2.2 Switch on the CETAC M-7500.

12.2.3 Verify the computer is on. If not, switch on the computer and enter network password and click on Start.

12.2.4 Go to programs, CETAC QuickTrace, and click on QuickTrace.

12.2.5 To create a new worksheet from a Template, Click on File, New From. Click on the browse button to select a Template worksheet, then enter the new File name (ex. Naming file 0713111AJT denotes the month, date, year, and analyst. The number at the end of the each run within a day's output.). Click Save. Click ok.

12.2.6 To open an existing worksheet, select Open, and select the desired worksheet. The worksheet will open to the sequence page.

12.2.7 To enter sample information, go to the sequence page. Follow the Template to fill in Ids for the Calibration QC and samples, under the sample label column

12.2.8 A typical sequence will consist of the following in order: a calibration curve (5 standards plus a blank), an ICV immediately followed by an ICB, a CRDL, a CCV immediately followed by a CCB. At this point a batch consisting of samples and batch QC can be analyzed. At a maximum of every 10 batch injections a CCV immediately followed by a CCB must be analyzed. The batch must also end with CCV immediately followed by a CCB. For 7471B, a CRDL must bracket the samples.

12.2.9 Go to File, Save to ensure changes to the worksheet are saved.

In the Method Editor Page the Conditions are set at:

Gas Flow (mL/min)	100
Pump Speed (%)	50
Sipper Depth (mm)	145
Sample Uptake (s)	35
Rinse Time (s)	95
Read Delay Time (s)	Varies with peak profile.
Replicate Read Time (s)	1.5
Replicates	4

12.2.10 Under Method editor, click QC Tests to Set the QC concentration and the control limits.

- 12.2.11 On the Sequence Parameters page hit the control button to set up when the QC will be analyzed, i.e. after calibration, after 10 samples, and at the end. IF re-calibration occurs the QC must be analyzed at the same frequency. This page also allows you to put the system into standby mode. The options allow you turn the pump on “slow” or “off. It also allows you to turn the Lamp off and Gas off.
- 12.2.12 The Sequence Parameters under the reports button allows you to customize the report printout by selecting solution information, report contents, and default number format.
- 12.2.13 Under the Sequence Editor page click on Manual QC, specify what to do if calibration QC fails, for example stop analysis, flag and continue, repeat-flag and continue, recalibrate and repeat, recalibrate and repeat with samples, or reslope and repeat.
- 12.2.14 On the Auto QC page of the Sequence Editor, specify what QC to run at the end of a run and corrective action if QC fails
- 12.2.15 Under the Sequence Editor click Sequence... This is where you determine the number of samples in the analysis and how often calibrations will be performed.
- 12.2.16 The analysis screen is accessed by clicking on the analysis button, from the main toolbar. This screen is where analysis controls and displays are located.

12.3 Starting the Analysis

- 12.3.1 Prepare the required reagents: Acid Rinse solution and reducing agent.
- 12.3.2 Turn on the lamp and carrier gas. A minimum 15 minute warm-up time is required.
- 12.3.3 Put peristaltic tubing in place, and clamp in place.
- 12.3.4 Place the auto-sampler rinse tubing into the Acid Rinse bottle. If rinse pump is not on make sure it's on by clicking on the instrument page and clicking “pump on”. Make sure to have probe down at this time as well.
- 12.3.5 Place the SnCl₂ line in a bottle of reagent water and start the peristaltic pump. Inspect flow to make sure lines are flowing correctly and not pulsing.
- 12.3.6 Wet the GLS (Gas Liquid Separator) center post. In the software click on the instrument icon, click analyzer, set gas flow to 350-mL/min and change pump speed to 100%. Pinch the drain line until 2 or 3 bubbles go to the top of the GLS center post. Then release the drain line and allow liquid to restore itself.
- 12.3.7 Attach GLS exhaust tube to GLS center post and close the optical cabinet door. Place reagent capillaries in appropriate reagent bottles.

- 12.3.8 Open the appropriate worksheet and verify that the gas flow and pump speed in the worksheet matches what is listed in instrument/analyzer, if the flow and the speed is not the same make the necessary change or click the auto set icon on the menu bar. This will stabilize the instrument before auto-zeroing and running a peak profile.
- 12.3.9 Record the Lamps mA's in a daily instrument logbook by clicking on the instrument icon, clicking on analyzer, "status of lamp".
- 12.3.10 Peak profile the high standard of the calibration. To do this click on Method Editor, read a sample icon, and then choose the location of high standard. Record the concentration of the peak profile standard in a daily instrument logbook. The read delay time is adjusted here as well.
- 12.3.11 Hit the GO icon to start the calibration. Once the calibration is complete a dialog window will generate stating, "Continue with Analysis" Click YES if satisfied with Calibration or click NO to re-analyze Calibration.
- 12.3.12 Hit the STOP icon to immediately end an analysis that is currently running. The auto-sampler probe will immediately return to the rinse station. Stopping an analysis in progress will prevent data from being saved for that sample.
- 12.3.13 To stop the analysis after the current sample select the Stop/After solution item from the analyze menu.
- Note: If you stop during sample uptake, or before the rinse has been completed, make sure you allow sufficient time in the rinse station before re-starting the analysis.**
- 12.3.14 If you stopped the analysis, you can simply restart by clicking the GO button. The analysis will begin at the next un-analyzed solution within the sequence.
- 12.3.15 To restart the analysis from the beginning you will need to generate the sequence again. Open the Sequence Editor, make any desired changes, click on Generate Sequence. Then click GO to start analysis over.
- 12.3.16 To read a single sample, click on the icon, analyze single sample. A dialog window will be brought up where you will enter the tube position, sample label, sample type, and other information if desired.
- Note: Using the read sample feature may invalidate your QC setup. If you have stopped during a batch analysis to read a single sample, it will be inserted into the sequence after the last analyzed sample.**
- 12.3.17 When the run is complete the instrument will automatically go into standby mode. Depending what options you have selected from sequence parameters, this may turn off or slow the pump, turn off the lamp, and/or the carrier gas flow.
- 12.3.18 To export the data, go to File then Export. Go to K:\Metals\CVAAS\40HG2\, enter file name, and click Save. .

12.4 System Shutdown

- 12.4.1 Place the inlet of the SnCl₂ line in a container of 10% nitric acid rinse (use a 500mL container; use 50mL of Nitric acid and dilute to 500 with nanopure water)) for 10 minutes.
- 12.4.2 Once the SnCl₂ is rinsed with 10% nitric acid rinse, place the SnCl₂ line into DI water for 1 minute to rinse system.
- 12.4.3 Remove the SnCl₂ line from the DI water and raise lines out of rinse solution. Run the pump until the lines are dry.
- 12.4.4 Raise the probe from the rinse station by clicking on the instrument icon and by clicking the Move Sipper Up button. Turn off peristaltic pump by clicking the Pump Off button.
- 12.4.5 Release all four peristaltic pump channel clamps and remove the tubing from the channel.
- 12.4.6 Remove the GLS exhaust tube from the GLS center post.
- 12.4.7 Turn off the gas and lamp by clicking on the instrument icon, then click analyzer. Turn the Lamp Off and set the gas to 0.
- 12.4.8 Close out of the software

13. Quality Control

- 13.1 Calibration Blanks – Calibration blanks (ICB and CCB) may not contain concentrations in excess of the LOQ. If blank results are not acceptable, analysis of the sequence should be halted. Corrective actions could include preparation of fresh calibration standards and blanks.
- 13.2 Calibration Checks (ICV and CCV)
 - 13.2.1 For 7470A, 7471M and 7471B the ICV and CCV limits are $\pm 10\%$ of their expected values. If outside the limits, reanalyze once. If still outside the limits, recalibrate.
 - 13.2.2 For 245.1 the ICV acceptance limits are $\pm 5\%$ and the CCV limits are $\pm 10\%$ of their expected values. If outside the limits, reanalyze once. If still outside the limits, recalibrate.
- 13.3 Pace Reporting Limit Standard (PRL or CRDL Std.)
 - 13.3.1 A standard prepared at the concentration of the lowest calibration point. It is analyzed after the ICV/ICB. When analyzing by 7471B the CRDL must also bracket all samples.

- 13.3.2 The CRDL must recover from 70-130% of true value for 7471B. The rest of the methods must be 60-140% recovery. If outside the limits, reanalyze once. If still outside the limits, recalibrate.
- 13.4 Batch – A preparation (digestion) batch will consist of up to 20 samples.
- 13.5 Laboratory Control Spike (LCS)
 - 13.5.1 A Laboratory Control Spike (LCS) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent.
 - 13.5.2 A Laboratory Control Spike Duplicate (LCSD) is performed if there is insufficient sample available for a MS/MSD or if requested by the client.
 - 13.5.3 For methods 7470A and 7471B the acceptance criterion is 85-115%. For method 245.1 the acceptance criterion is 70-130% recovery. For method 7471BM the acceptance criterion is based on historical.
 - 13.5.4 If the LCS and or LCSD are outside of acceptance criterion, then all the samples prepared in the batch must be re-prepped and re-analyzed.
 - 13.5.5 When a LCSD is included, the acceptance criterion for precision is 20% RPD. If insufficient sample remains to re-prepare and re-analyze, the data qualifiers is given to all associated samples.
- 13.6 Method Blank (MB)
 - 13.6.1 A MB must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent.
 - 13.6.2 The MB must not contain mercury at a concentration at or above the LOQ.
 - 13.6.3 Any samples digested with an unacceptable method blank must be re-prepped and analyzed unless the sample concentrations are less than the level being reported at or more than 10 times the value found in the method blank.
 - 13.6.4 In those cases that LOD reporting is required, the MB must be evaluated to the LOD. For LOD reporting, an appropriate data qualifier is given to samples associated with \pm MB hits between the \pm LOD and \pm LOQ where the sample results are less than 10 times the value found in the method blank.
 - 13.6.5 For negative instrument measurements $>$ LOD and $<$ LOQ qualify sample results that are non-detections and $<$ 10 times the measurement with “Analyte was measured in the associated method blank at a concentration of -#.# units.” Make sure to enter the concentration and applicable sample units.
- 13.7 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

- 13.7.1 A MS/MSD pair must be prepared and analyzed for each batch or every 20 samples that are similar in matrix at a frequency of 5% for 7470A and 7471B methods and 10% for 245.1.
- 13.7.2 For TCLP, SPLP, and ASTM samples one Matrix Spike (MS) must be prepared per sample matrix (ie: soil and wood chips would each require a separate matrix spike).
- 13.7.3 The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip, or equipment blanks can be used for MS/MSD.
- 13.7.4 For methods 7470A and 7471B the acceptance criterion is 85-115%. For method 245.1 the acceptance criterion is 70-130%. For method 7471BM the acceptance criterion is generated from historical.
- 13.7.5 If one or both spike recoveries are outside recovery acceptance criterion, the parent sample and failing QC point are given an appropriate data qualifier.
- 13.7.6 If the precision is outside the 20% RPD criterion, the parent sample and QC point are given an appropriate data qualifier.
- 13.8 Duplicate Sample (DUP)
 - 13.8.1 Typically the method requirements for duplicate sample analysis are met with the MSD or LCSD, but based on client request a DUP may also be prepared and analyzed.
 - 13.8.2 The DUP is evaluated for precision with the parent sample. If the RPD is outside 20% RPD criterion the parent sample and DUP are given an appropriate data qualifier. Parent sample is chosen at random or assigned by the client.
- 13.9 For dissolved samples filtered in house a filter blank is created. The filter blank is evaluated and qualified the same as a method blank.
- 13.10 All reported results must be within the range of the calibration curve. Dilute when results are greater than the high standard in the curve.

14. Data Analysis and Calculations

- 14.1 **Water/TCLP/SPLP/ASTM Samples** – Since initial sample aliquot and final digestate volumes are the same, the mercury analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.
- 14.2 **Soil/Solid/Biota Samples** –
 - Final Result (mg/kg dry weight corrected) =
$$\frac{\text{Raw data result } (\mu\text{g/L}) * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$
 - Biota results can be reported on an as is/wet weight basis. The dry weight correction in the formula is then not applicable.

14.3 Wipes

$$\text{Final Result (Total } \mu\text{g)} = \frac{\text{Raw data result (} \mu\text{g/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{1 \text{ wipe}}$$

14.4 Accuracy-

$$\text{Spike Percent Recovery} = \frac{\text{Spike Sample Result} - \text{Sample Result}}{\text{Spike Added}} \times 100$$

14.5 Precision-

$$\text{Relative Percent Difference} = \frac{\text{Spike Sample Result} - \text{Spike Sample Duplicate Result}}{(\text{Spike Sample Result} + \text{Spike Sample Duplicate Result})/2} \times 100$$

15. Data Assessment and Acceptance Criterion for Quality Control Measures

Table 5
QC SUMMARY

Analytical Method ⇒ Calibration Measure ↓	EPA SW846 7470A, 7471B, 7471BM for Tissue, 245.1 Frequency	Acceptance Criterion
Laboratory Control Spike and Laboratory Control Spike Duplicate (LCS/LCSD)	<ul style="list-style-type: none"> One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent. A LCSD is required if MS/MSD is not performed or if requested by the client. 	<ul style="list-style-type: none"> Project Specific or For 245.1, 7470A, and 7471B 85 – 115% with 20% RPD For Biota by 7471BM Historical with 20% RPD
Matrix Spike / Matrix Spike Duplicate (MS/MSD)	<ul style="list-style-type: none"> One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. 	<ul style="list-style-type: none"> Project Specific or For 7470A, and 7471B 85 –115% with 20% RPD For 245.1 70-130% with 20% RPD For Biota by 7471M Historical with 20% RPD
Method Blank (MB)	<ul style="list-style-type: none"> One per batch of samples, up to 20 environmental samples, whichever is more frequent. 	<ul style="list-style-type: none"> Project Specific or Less than the LOQ
Initial Calibration	<ul style="list-style-type: none"> Analyzed daily before samples Minimum 5 standards plus a blank 	<ul style="list-style-type: none"> Correlation coefficient must be 0.995 or greater
Initial Calibration Verification (ICV)	<ul style="list-style-type: none"> Analyzed after calibration. 	<ul style="list-style-type: none"> 245.1 recovery must be between 95 – 105% 7470A and 7471B recovery must be between 90 – 110%
Initial Calibration Blank (ICB)	<ul style="list-style-type: none"> Analyzed after ICV. 	<ul style="list-style-type: none"> Project specific or Less than LOQ
CRDL	<ul style="list-style-type: none"> 7470A, 7471M Analyzed after Initial calibration blank. 7471B – At a minimum, CRDLs must bracket all samples reported by this method. 	<ul style="list-style-type: none"> At the lowest calibration point 7471B 70-130% 7470A, 245.1, 7471M 60-140%
Continuing Calibration Verification (CCV)	<ul style="list-style-type: none"> Analyzed after every 10 samples. 	<ul style="list-style-type: none"> Project specific or Recovery between 90 – 110%
Continuing Calibration Blank (CCB)	<ul style="list-style-type: none"> After each CCV. 	<ul style="list-style-type: none"> Project specific or Less than LOQ

16. Corrective Actions for Out-Of-Control Data

Table 6

DATA ASSESSMENT/CORRECTIVE ACTION

Analytical Method Acceptance Criterion ⇒ Data Assessment Measure ↓	Method Citation: EPA SW846 7470, 7471, 245.1, 7471M for Tissue If these conditions are not achieved ⇒
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
Initial / Continuing Calibration Verification	• 5
Initial / Continuing Calibration Blank	• 6
Holding Time Compliance	• 7
CRDL	• 8

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.
2. In the absence of project specific or method requirements, in-house generated limits will be used. If the MS or MSD fail because the concentration of the spike is less than 25% of the concentration of the parent, use appropriate flag for the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, use appropriate flag for the parent sample if either the MS and/or MSD fail. If the MS and MSD fail precision control limits flag the parent with the appropriate precision data qualifier.
3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and/or appropriate precision qualifier to reflect the deficiencies. Generate a Non-Conformance Memo.
4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.
5. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.
6. If ICB/CCB is outside the control limits, reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB do not have to be reanalyzed or qualified.
7. Flag results with the appropriate data qualifier.
8. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

- 17.1 See Section 16, Table 6: Corrective Action.

18. Method Performance

- 18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.
- 18.2 The analyst must read and understand this procedure with written documentation maintained in his/her training file.
- 18.3 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures* (current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager. A continuing demonstration of capability (CDOC) must be performed annually.
- 18.4 A linear dynamic range (LDR) study must be performed yearly. The range tested will be several factors greater than the calibration curve, but will not be extended until failure so as to not harm the instrument with high concentrations of mercury. A passing level must have a recovery within 10% of the standard being analyzed.
- 18.5 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of the LOD and LOQ* (current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.
- 18.6 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program* (current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office. At a minimum, these are performed twice a year for the aqueous and soil matrices.

19. Method Modifications

- 19.1 For EPA 245.1, the lab will digest the calibration curve consistent with SW846 7470A requirements. The volumes and concentrations used to make the calibration curve deviate from 7470A and 7471B. The methods are based on using 300 mL BOD bottles that are no longer currently used. The resulting calibration curve encompasses the same working range for reporting data.
- 19.2 EPA 245.1 and EPA 245.6 both have the stannous chloride solution being made up with 25 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ to a final volume of 250 mL with 0.5 N H_2SO_4 . EPA SW846 7470A and 7471B both have stannous sulfate being made up the same way with the option of using stannous chloride in place of the stannous sulfate. EPA SW846 7471B mentions making the stannous sulfate in water. The lab uses stannous chloride made from 100 g of SnCl_2 and 70 mL of concentrated HCl diluted to a final volume of 1,000 mL.

- 19.3 For SW846 7470A and EPA 245.1, the lab has chosen to use a 10mL sample volume instead of the method specified 100mL. All standards and reagents have been reduced to ensure that the ratios are still consistent with the method.
- 19.4 For SW846 7471A/B, the lab has chosen to use a 0.3g sample volume instead of the method specified 0.5-0.6g. All standards and reagents have been reduced to ensure that the ratios are still consistent with the method.
- 19.5 SW846 7471A/B has been modified to use in the digestion and analysis of biological tissue samples. Sample volume, reagents and standards have not been altered.

20. Instrument/Equipment Maintenance

- 20.1 Any daily or periodic maintenance must be recorded in the instrument maintenance logbook.

21. Troubleshooting

- 21.1 Please see the instrument manual for information on instrument troubleshooting.

22. Safety

- 22.1 Standards and Reagents – The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses.
- 22.2 Safety Data Sheets (SDSs) – A reference file of SDS are on file in the laboratory and available to all personnel. A formal safety plan has been prepared and distributed to all personnel with documented training.
- 22.3 Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood.
- 22.4 Samples – Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

Note: Extreme caution must be used when preparing rodents for digestion. The samples must undergo a special procedure to destroy any Hantavirus, which may be present. Refer to the most recent version of SOP S-GB-L-002 *Small Rodent Handling and Homogenization* for details.

- 22.5 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the SDS sheets for all chemicals and reagents they use for this procedure and the location of the SDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

23. Waste Management

- 23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management*.

24. Pollution Prevention

- 24.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

25. References

- 25.1 EPA Method 245.1 Revision 3.0
- 25.2 SW-846 7470A, Revision 1, September 1994
- 25.3 SW-846 7471A, Revision 1, September 1994
- 25.4 SW-846 7471B, Revision 2, February 2007
- 25.5 PASI Quality Manual, current revision
- 25.6 National Environmental Laboratory Accreditation Conference (NELAC), July 2003 Standards.
- 25.7 The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version

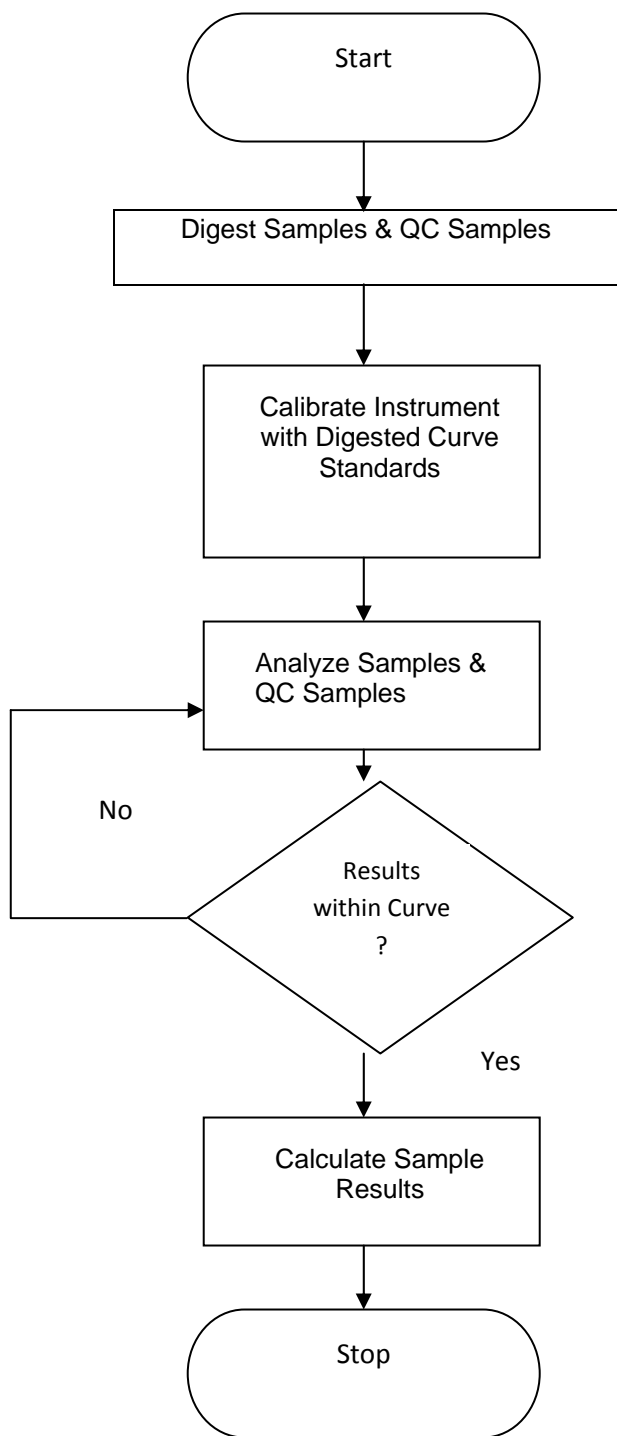
26. Tables, Diagrams, Flowcharts, and Validation data

- 26.1 Mercury Process Flow Chart

27. Revisions

Document Number	Reason for Change	Date
S-GB-M-017-Rev.03	Section 12.2: Solid digestion has been updated to include the heating step and increase the acid ratios. Throughout document: Included uncontrolled document disclaimer..	26Jun2015
S-GB-M-017-Rev.04	Cover page: Updated QM name. Section 7.4.2: Added field filter information. Section 7.5: Updated leach sample information. Section 9.4: Deleted balance since also listed in Section 9.15. Section 10: Updated to include expiration dates. Section 10.14: Diluent added. Table 3: Changed to Table for 7471B Solids and Tissue. Table 4: Changed to Table for 7470 and 245.1 standards. Table 5: Deleted. Section 11.1.1.7: Changed spike to 1000ug/L at 0.05mL. Section 12: Changed spike solution volumes as needed.. Section 12.1.3.12, 12.1.3.13 and 12.1.3.15: Added. Section 12.2: Entire Section re-wrote to match current practice. Section 13 and Table 5: Added QC requirements and updated table. Section 18.4: Added LDR requirement. Section 19: Updated method modifications. Throughout Document: Changed digestion temperature range from 95±5°C to 95±3°C.	13Jun2016

Mercury Process Flow Chart




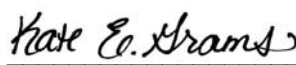

STANDARD OPERATING PROCEDURE

Analysis of Organochlorine Pesticides by Gas Chromatography

Reference Methods: SW-846 8081A/B/ EPA 608

SOP NUMBER	S-GB-O-027-Rev.08
EFFECTIVE DATE	Date of Final Signature
SUPERSEDES	S-GB-O-027-Rev.07

APPROVAL

 _____ Nils Melberg, Laboratory General Manager	_____ Date
 _____ Kate Grams, Laboratory Quality Manager	_____ Date
 _____ Chris Haase, Department Manager	_____ Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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S-GB-O-027-REV.08

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1. PURPOSE/IDENTIFICATION OF METHOD

- 1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the analysis of various organochlorine pesticides in water, soils, sediments, biological tissue, and solid waste compliant with SW-846 Method 8081A/B and EPA 608. Samples for analysis are prepared by SW846 Method 3510C, 3540C, 3541, and 3550B. See Section 25 for list of reference SOPs.

2. SUMMARY OF METHOD

- 2.1 A volume of sample extract in hexane is injected into a gas chromatograph (GC) and the compounds in the GC effluent are detected by an electron capture detector (ECD) and then analyzed.

3. SCOPE AND APPLICATION

- 3.1 Personnel: This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/electron capture detection (GC/ECD) systems and interpretation of complex chromatograms. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.
- 3.2 Parameters: This method is used to determine the concentration of the various organochlorine pesticides listed in Section 5, Table 1: Analyte List and Reporting Limits, utilizing a gas chromatograph equipped with dual electron capture detectors. Reporting limits are subject to change based on current analytical system performance and actual sample matrices.

4. APPLICABLE MATRICES

- 4.1 This SOP is applicable to the analysis of organochlorine pesticides in aqueous, soils, solids, domestic and industrial wastes and biological samples.

5. LIMITS OF DETECTION AND QUANTITATION

5.1 The reporting limits (PQL) of this method for Organochlorine Pesticides are listed in Table 1 below. All current MDLs are listed in the LIMS and are available by request from the Quality Department.

Table 1: Analyte List and Reporting Limits

Compound	CAS #	Aqueous PQL (µg/L)	Solid PQL (µg/Kg)	Biota PQL (µg/Kg)
2,4'-DDD (o,p'-DDD)	53-19-0	0.05	1.7	2.5
2,4'-DDE (o,p'-DDE)	3424-82-6	0.05	1.7	2.5
2,4'-DDT (o,p'-DDT)	789-02-6	0.05	1.7	2.5
4,4'-DDD (p,p'-DDD)	72-54-8	0.10	3.3	5.0
4,4'-DDE (p,p'-DDE)	72-55-9	0.10	3.3	5.0
4,4'-DDT (p,p'-DDT)	50-29-3	0.10	3.3	5.0
Aldrin	309-00-2	0.050	1.7	2.5
alpha-BHC	319-84-6	0.050	1.7	2.5
alpha-Chlordane	5103-71-9	0.050	1.7	2.5
beta-BHC	319-85-7	0.050	1.7	2.5
cis-Nonachlor	5103-73-1	0.05	1.7	2.5
Delta-BHC	319-86-8	0.050	1.7	2.5
Dieldrin	60-57-1	0.10	3.3	5.0
Endosulfan I	959-98-98	0.050	1.7	2.5
Endosulfan II	33213-65-9	0.10	3.3	5.0
Endosulfan Sulfate	1031-07-8	0.10	3.3	5.0
Endrin	72-20-8	0.10	3.3	5.0
Endrin Aldehyde	7421-93-4	0.10	3.3	5.0
Endrin Ketone	53494-70-5	0.10	3.3	5.0
gamma-BHC (Lindane)	58-89-9	0.050	1.7	2.5
gamma-Chlordane	5103-74-2	0.050	1.7	2.5
Heptachlor	76-44-8	0.050	1.7	2.5
Heptachlor Epoxide	1024-57-3	0.050	1.7	2.5
Hexachlorobenzene	118-74-1	0.050	1.7	2.5
Methoxychlor	72-43-5	0.50	17	25
Mirex	2385-85-5	0.05	1.7	2.5
Oxychlordane	27304-13-8	0.05	1.7	2.5
Pentachloroanisole	1825-21-4	0.050	1.7	2.5
trans-Nonachlor	39765-80-5	0.05	1.7	2.5
Toxaphene	8001-35-2	3.0	100	150
Technical chlordane	57-74-9	1.0	33	50

6. INTERFERENCES

- 6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware. These interferences lead to discrete artifacts or elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences by running reagent blanks and method blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates, which are easily extracted during laboratory operations. Avoiding the use of such plastics in the laboratory can best minimize interferences from phthalates.
- 6.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. Cleanup procedures such as Gel Permeation Chromatography (GPC), Florisil cartridge cleanup, Silica Gel Separation, and sulfur removal are available for the most common interference's encountered. These cleanup procedures are described separately in the most current revision or replacement of the laboratory SOPs: S-GB-O-032 *Gel Permeation Chromatography Clean-up by SW846 3640A*, S-GB-O-037, *Florisil Cartridge Clean-up for Organochlorine Pesticide Samples*, S-GB-O-038, *Silica Gel Clean-up for Organic Analysis* and S-GB-O-039 *Copper Clean-up for the Removal of Sulfur from PCB and Toxaphene Samples*.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Table 2: Sample Collection, Preservation, Storage and Hold Time

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One 1L amber glass	None	≤6°C	7 days
Soil/Solid (non-aqueous)	One 8oz wide glass amber jar	None	≤6°C	14 days
Biological Tissue	--	None	≤ -10°C	365 days or longer per client request prior to extraction, typical extraction hold time do not apply.
TCLP/SPLP	One 1L amber glass	None	≤6°C	14 days to leach, 7 days to for solvent extraction after the tumbling process has occurred.
Extracts	ONE 5 or 10mL glass amber vial.	None	≤6°C	40 days

8. DEFINITIONS

- 8.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1 Instrumentation

9.1.1 Gas Chromatograph: HP5890, HP6890 or HP7890 GC with dual electron capture detectors (or equivalent). The following are the gas chromatographic analytical conditions:

Table 3: Recommended Gas Chromatograph Operating Conditions

Operating Condition	40GCS6; HP5890	40GCSG; HP7890
Carrier Gas:	Helium 2.0 mL/min flow rate	Helium 2.0 mL/min flow rate
Make-up Gas:	Nitrogen 65 mL/min flow rate	Nitrogen 65 mL/min flow rate
Detector Temperature:	300°C	300°C
Injector Temperature:	200°C	200°C
Injection:	Splitless	Splitless
Injection Volume:	2 µL	2 µL
Initial Temperature:	180°C	130°C
Initial Hold Time:	1.0 min	0.5 min
Temperature Ramp:	20°C/min to 220°C, hold 1.0 min., 3°C/min to 226°C, hold 1.0 min, 20°C/min to 300°C, hold 2.8 min	20°C/min to 200°C, hold 0.0 min., 10°C/min to 230°C, hold 0.0 min, 20°C/min to 310°C, hold 3.0 min
Total Run Time:	13.5 min.	14.0 min.

9.1.2 GC Autosampler: HP7673A or HP 7693

9.1.3 Detector: ECD

9.1.4 Gas Chromatograph Columns: RTX-CLP, 30 m x 0.32 mm ID, .25µm film thickness (Restek or equivalent) and a RTX-CLP2, 30 m x 0.32 mm ID, .25 µm film thickness, (Restek, or equivalent). Other analytical columns may be used based on projects specific requirements. Columns are mounted in a dual GC/ECD with a single injection port/guard column connected to a glass Y-splitter.

9.1.5 Data Processor: ChemStation/HP Target

9.2 Glassware and Materials

9.2.1 Gastight Syringes – any size ranging from 10µL to 1000µL (Hamilton series 1000 or equivalent)

9.2.2 Autosampler Vials: 2 mL glass vials with crimp top caps.

9.2.3 Helium gas – Airgas Ultra high purity or equivalent

9.2.4 Nitrogen gas – Airgas Ultra high purity or equivalent

10. REAGENTS AND STANDARDS

10.1 Solvents – Hexane and acetone, pesticide grade.

Table 4: Solvents

Reagent	Purity	Manufacturer	Vendor	Catalog #	Expiration Date
Hexane	NS Grade	Burdick & Jackson	MG Scientific	B&J-217-4	Manufacturer Exp date or 2 year from receipt
Acetone	Pesticide Grade	Burdick & Jackson	MG Scientific	B&J-010-4	Manufacturer Exp date or 2 year from receipt

- 10.2 Stock Standard Solutions: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf life of standard solutions is 1 year from the date of preparation or the expiration date on the vendor label; whichever is earliest (see Table 5 for stock standards).
- 10.3 Calibration Standards: Single component standards are prepared from stock standard solutions at 5 concentration levels. One of the concentration levels should be at a concentration near, but above the method detection limit. Separate calibration standards are required for each multi-component target analyte (e.g. Toxaphene and Technical Chlordane). Shelf life of the calibration standards is 6 months from the date of preparation or the expiration date of the stock standard solution; whichever is earliest (see Table 6 for standard preparation.).
- 10.4 Surrogate Standards: Surrogate standards are used to monitor the performance of the method. They are added to all calibration standards. Shelf life of the surrogate standards is 6 months from the date of preparation or the expiration date of the stock standard solution; whichever is earliest (see Tables 5 and 6).
- 10.5 Performance Evaluation Mixture (PEM): A standard comprised of DDT and Endrin, which is analyzed at the beginning of every 12 hours to determine compound breakdown (see Tables 5 and 6).

Table 5: Stock Standards

Standard	Stock Standard	Conc.	Amount Used	Final Volume	Solvent Used	Final Conc.	Expiration Date
TCMX/DCB Stock	Restek Pesticide Surrogate Mix	200 µg/mL	1.0 mL	20 mL	Hexane	10 µg/mL	Manufacturer's recommended expiration date for unopened ampulated standards. 1 year after ampule is opened or on expiration date, whichever is sooner.
Pesticide A/B Stock	Restek Organochlorine A/B Mix	8-80 µg/mL	1.0	10 mL	-	0.8-8.0µg/mL	
Pesticide C-Mix Stock	O2Si	100µg/mL	1.0	50 mL	-	2.0µg/mL	
Tech. Chlordane Stock Solution	Restek Chlordane Mix	1000µg/mL	1.0	10 mL	-	100 µg/mL	
Toxaphene Stock	Restek Toxaphene Mix	1000µg/mL	1.0 mL	10 mL	Hexane	100 µg/mL	
Pesticide A/B ICV Stock	O2Si	0.4-4.0µg/mL	10.0 mL	200 mL	Hexane	0.4-4.0 µg/mL	
Pesticide C-Mix ICV Stock	Absolute Stds	100 µg/mL	1.0 mL	50 mL	Hexane	2.0 µg/mL	
Different Lot # than Calibration Std	Restek Toxaphene Mix	1000µg/mL	1.0 mL	10 mL	Hexane	100 µg/mL	
PEM Stock Solution	O2Si	200µg/mL	1.0 mL	20 mL	Hexane	10 µg/mL	

o2si and Restek Pesticide A/B contains alpha-BHC, beta-BHC, delta-BHC, gamma-BHC, Aldrin, Heptachlor, Heptachlor epoxide, Endosulfan I, Endosulfan II, Dieldrin, Endrin, Endrin aldehyde, Endosulfan sulfate, and Endrin ketone, 4,4'-DDE, 4,4'-DDD, 4,4'-DDT alpha-chlordane, gamma-chlordane, and Methoxychlor.

Pesticide C-Mix contains 2,4'-DDT, 2,4'-DDD, 2,4'-DDE, Hexachlorobenzene, Pentachloroanisole, Oxychlordane, trans-Nonachlor, cis-Nonachlor, Mirex and Isodrin. Isodrin is not a reported compound.

Table 6: Preparation of Analytical Standard Solutions

Standard	Pesticide Stock Standard	Amount Used (µL)	Final Conc. (µg/mL)	Surrogate Stock Standard	Amount Used (µL)	Final Conc. (µg/mL)	Solvent Used	Final Volume (mL)	Expiration Date
PEST-1 & RLVS	Pesticide A/B Stock	250	0.0050-0.050	TCMX/DCB Stock	40	0.010	Hexane	40	6 months from preparation or the expiration date listed for the stock source, whichever is sooner.
PEST-2	Pesticide A/B Stock	250	0.010-0.10	TCMX/DCB Stock	40	0.020	Hexane	20	
PEST-3	Pesticide A/B Stock	500	0.020-0.20	TCMX/DCB Stock	100	0.050	Hexane	20	
PEST-4	Pesticide A/B Stock	1000	0.040-0.40	TCMX/DCB Stock	200	0.10	Hexane	20	
PEST-5	Pesticide A/B Stock	1000	0.080-0.80	TCMX/DCB Stock	120	0.12	Hexane	10	
PEST-4 ICV	Pesticide A/B ICV Stock	1000	0.040-0.40	TCMX/DCB Stock	100	0.1	Hexane	10	
INDC-1 & RLVS	Pesticide C-Mix Stock	50	0.005	TCMX/DCB Stock	20	0.010	Hexane	20	

Standard	Pesticide Stock Standard	Amount Used (µL)	Final Conc. (µg/mL)	Surrogate Stock Standard	Amount Used (µL)	Final Conc. (µg/mL)	Solvent Used	Final Volume (mL)	Expiration Date
INDC-2	Pesticide C-Mix Stock	100	0.010	TCMX/DCB Stock	40	0.020	Hexane	20	6 months from preparation or the expiration date listed for the stock source, whichever is sooner.
INDC-3	Pesticide C-Mix Stock	200	0.020	TCMX/DCB Stock	100	0.050	Hexane	20	
INDC-4	Pesticide C-Mix Stock	400	0.004	TCMX/DCB Stock	200	0.10	Hexane	20	
INDC-5	Pesticide C-Mix Stock	800	0.080	TCMX/DCB Stock	240	0.12	Hexane	20	
INDC-4 ICV	Pesticide C-Mix ICV Stock	400	0.040	TCMX/DCB Stock	200	0.1	Hexane	20	
TOX-1 & RLVS	Toxaphene Stock	75	0.3	TCMX/DCB Stock	12.5	0.005	Hexane	25	
TOX-2	Toxaphene Stock	125	0.5	TCMX/DCB Stock	25	0.01	Hexane	25	
TOX-3	Toxaphene Stock	375	1.5	TCMX/DCB Stock	50	0.02	Hexane	25	
*TOX-4	Toxaphene Stock	1200	3.0	TCMX/DCB Stock	200	0.05	Hexane	40	
TOX-5	Toxaphene Stock	1000	4.0	TCMX/DCB Stock	250	0.10	Hexane	25	
TOX-6	Toxaphene Stock	1250	5.0	TCMX/DCB Stock	375	0.15	Hexane	25	
TOX-4 ICV	Toxaphene ICV Stock	1200	3.0	TCMX/DCB Stock	200	0.050	Hexane	40	
TCHLOR-1 & RLVS	Technical Chlordane Stock	10	0.10	TCMX/DCB Stock	10	0.010	Hexane	10	
TCHLOR-2	Technical Chlordane Stock	20	0.20	TCMX/DCB Stock	20	0.020	Hexane	10	
TCHLOR-3	Technical Chlordane Stock	40	0.40	TCMX/DCB Stock	50	0.050	Hexane	10	
*TCHLOR-4	Technical Chlordane Stock	80	0.80	TCMX/DCB Stock	100	0.10	Hexane	10	
TCHLOR-5	Technical Chlordane Stock	100	1.0	TCMX/DCB Stock	120	0.12	Hexane	10	
PEM Working Standard	PEM Stock Solution	0.6 mL	0.15	NA	NA	NA	Hexane	40	

* Level used in 1 point calibration of multicomponent.

11. CALIBRATION AND STANDARDIZATION

- 11.1 Prime (or deactivate) the column by injecting a pesticide standard mixture at the high point in the calibration curve. Inject this standard mixture prior to beginning the initial calibration or calibration verification at the beginning of the analytical sequence.
- 11.2 A solvent blank may be run following the prime to verify there is no carryover or contamination introduced from the injection process. Solvent blanks may be placed into the analytical sequence at any position by the analyst to demonstrate that the analytical system is not contaminated. Solvent blanks, however, may not be routinely placed immediately prior to continuing calibration verification standards, PEM's, etc.
- 11.3 A Performance Evaluation Mixture (PEM), a standard comprised of DDT and Endrin, will be analyzed before samples are analyzed and at the beginning of every 12 hours for all methods except EPA 608. If the breakdown for either compound exceeds 15%, corrective action must be taken prior to calibration. Breakdown is calculated by the following equations:

$$\% \text{ Breakdown DDT} = \frac{\text{Response (DDD + DDE)}}{\text{Response (DDD + DDE + DDT)}} * 100$$

$$\% \text{ Breakdown Endrin} = \frac{\text{Response (Endrin aldehyde + Endrin Ketone)}}{\text{Response (Endrin + Endrin aldehyde + Endrin Ketone)}} * 100$$

- 11.3.1 The PEM standard is not analyzed when Toxaphene is the only analyte being quantified and reported from the analysis.
- 11.4 Initial Calibration: The initial calibration includes the analysis of five concentrations of each single component pesticide and surrogate compound and a single point calibration for each multi-component analyte (unless otherwise necessary for a specific project). See SW-846 Method 8000B for additional guidelines on proper initial calibration and calibration verification.
- 11.4.1 The initial calibration sequence contains the following injections that must be analyzed before any samples. (Sequence subject to change):
1. PEM
 2. Toxaphene – LVL 4
 3. T-Chlordane – LVL 4
 4. Pesticide A/B Mix #5
 5. Pesticide A/B Mix #4
 6. Pesticide A/B Mix #3
 7. Pesticide A/B Mix #2
 8. Pesticide A/B Mix #1
 9. Pesticide C Mix #1
 10. Pesticide C Mix #2
 11. Pesticide C Mix #3
 12. Pesticide C Mix #4
 13. Pesticide C Mix #5
 14. Hexane
 15. PEM
 16. CRDL AB (RVLS)
 17. CRDL C (RVLS)
 18. Initial Calibration Verification –A/B Mix
 19. Initial Calibration Verification – C Mix

11.5 Initial Calibration Acceptance Criteria

- 11.5.1 Linear Calibration using Average Response Factors: Calculate the response factor (RF) for each analyte at each concentration, the mean response factor, and the relative standard deviation (RSD) of the response factors using the formulas below.

$$\begin{aligned}\text{Response Factor (RF)} &= \frac{\text{Peak Area}}{\text{Standard concentration } (\mu\text{g/mL})} \\ \text{\%RSD} &= \frac{\text{Standard Deviation}}{\text{Average Response Factor}}\end{aligned}$$

- 11.5.2 For SW846 8081A/B: Any target analyte which has calibration factors with a relative standard deviation (RSD) of less than 20% is considered valid and the average response factor may be used for quantitation purposes. If the %RSD exceeds 20%, the analyst must use linear regression for determining analyte concentrations.
- 11.5.3 For EPA 608: Any target analyte which has calibration factors with a relative standard deviation (RSD) of less than 10% is considered valid and the average response factor may be used for quantitation purposes. If the %RSD exceeds 10%, the analyst must use linear regression for determining analyte concentrations.
- 11.5.4 If linear regression is used, the intercept should not be forced through the origin. The regression calculation will generate a correlation coefficient “r”. In order to be used for quantitative purposes the r-value must be greater than 0.99.
- 11.5.5 All initial calibration and calibration verification criteria apply to both analytical columns.
- 11.5.6 The initial calibration may continue to be used as long as the analytical system meets acceptance criteria. If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. Perform instrument maintenance as necessary and recalibrate the instrument.

11.6 Initial Calibration Verification

- 11.6.1 To ensure the calibration standards are at the correct concentration, an initial calibration verification (ICV) will be analyzed against the initial calibration curve prior to the analysis of samples. The ICV is a midpoint of the Pesticide A/B Mix, (and Pesticide C Mix if included in the initial calibration) and must be from a different source or from a different lot number from the same vendor.
- 11.6.1.1 For SW846 8081A: The difference between each individual compound response of the ICV and initial calibration standard must be within 15% of one another on the basis of each compound or the average across all compounds.
- 11.6.1.2 For SW846 8081B: The difference between each individual compound response of the ICV and initial calibration standard must be within 20% of one another.
- 11.6.1.3 For EPA 608: The difference between each individual compound response of the ICV and initial calibration standard must be within 15% of one another.

11.6.2 The injection of the first ICV begins the 12-hour clock in which samples may be analyzed.

11.7 Continuing Calibration Verification

11.7.1 Continuing Calibration Verification (CCV) will include the injection of the PEM standard, midpoint calibration checks of the Pesticide A/B Mix, and Pesticide C Mix if included in the sample analyte list.

11.7.2 A PEM and CCV must be analyzed every 12 hours or 20 samples, whichever is more frequent. It is recommended that calibration verification be performed after every 10 samples to minimize reruns due to calibration verification failure.

11.7.2.1 For SW846 8081A: The difference between each individual compound response of the CCV and initial calibration standard must be within 15% of one another on the basis of each compound or the average across all compounds.

11.7.2.2 For SW846 8081B: The difference between each individual compound response of the CCV and initial calibration standard must be within 20% of one another.

11.7.2.3 For EPA 608: The difference between each individual compound response of the CCV and initial calibration standard must be within 15% of one another.

11.7.3 Sample data is not acceptable unless bracketed by acceptable analyses of a CCV. Any samples not bracketed by an acceptable CCV must be reanalyzed. The ending PEM of a sequence may fail breakdown and samples can be reported.

11.8 Initial Calibration Verification and Continuing Calibration Verification Acceptance Criteria

11.8.1 The percent difference (%D) is determined for all analytes in the ICV/CCV. The individual compound %D must be within the above listed method criteria of the calibration curve for each analyte.

$$\%D = \frac{R_2 - R_1}{R_2} * 100$$

Where: R_1 = Theoretical Value
 R_2 = Calculated Amount

The analyst should verify that the software is using appropriate values for calculations.

11.8.2 If the acceptance criteria for the ICV is not met, inspect the gas chromatographic system for problems. Re-inject the standard; if acceptance criteria is not met, then a new initial calibration must be performed.

11.8.3 Results may be reported for CCV failures, biased high, if failed compounds are not detected in samples.

11.8.4 Any analyte, for any reason, not meeting the CCV acceptance criteria must be qualified, if reported to the client.

11.9 Retention Time Windows

11.9.1 Retention time windows are determined for all single and multi-components analytes.

11.9.1.1 Make at least four injections of all analytes of interest over a 72-hour period.

11.9.1.2 Record the retention time (RT) for each analyte, and selected peaks for multi-component analytes, to three decimal places. Calculate the mean and standard deviation for each peak.

11.9.1.3 The width of the retention time window is defined as ± 3 standard deviations of the mean established. The minimum retention window will be ± 0.030 minutes.

11.9.1.4 Establish the center of the RT window for each analyte and surrogate using the absolute RT from the calibration verification standard at the beginning of the analytical shift. Optionally, the initial calibration retention time windows may continue to be used as long as the method criteria are met. For samples run during the same shift as an initial calibration, use the RT of the mid-point standard in the initial calibration.

11.9.1.5 RT windows must be calculated on each column and instrument when a new GC column is installed. Additional guidance is provided in SW846 8000B.

11.9.2 The retention time of each analyte must fall within its respective retention time window for all standards. If not, the gas chromatographic system must be evaluated. Re-inject the standard; if not acceptable then a new calibration must be performed.

11.10 Reporting Limit Verification Standard (RVLS) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter. Recovery must be 60-140% of true value to be accepted. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12. PROCEDURE

12.1 Sample Analysis

12.1.1 For SW846 8081A/B a matrix spike/matrix spike duplicate must be prepared and analyzed at least once for each matrix type per every twenty samples. For EPA 608 a matrix spike/matrix spike duplicate must be prepared and analyzed at least once for each matrix type per every ten samples. The sample use for the MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. If insufficient sample volume is available to perform a MS/MSD, a laboratory control spike and laboratory control spike duplicate will be used instead.

12.1.2 Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, matrix spike duplicates and laboratory control samples.

12.1.3 The laboratory will identify and quantify peaks based on RT and the average calibration factor established during the initial calibration sequence.

- 12.1.4 The PEM and Continuing calibration verification are analyzed every 20 samples (not to exceed 12 hours) or less during an analytical sequence in order to monitor retention times, calibration factors, and column performance. (It is recommended that calibration verification be performed after every 10 samples to minimize reruns due to calibration verification failure.) Sample data can be collected only as long as the results for this standard fall within the defined limits. If two consecutive unacceptable standards are run, all extracts run since the previous acceptable standards must be reanalyzed. The ending PEM of the sequence is allowed to fail.
- 12.1.5 Failure to meet any of the criteria established in this method must be thoroughly documented, and technical justification for the validity of the data must be presented to the section supervisor.
- 12.2 Quantitation of Analytes
- 12.2.1 Analytes must be quantified with a laboratory data system. Peak area is the basis for quantitation.
- 12.2.2 Quantitation of *single response pesticides* and surrogate standards is performed using the column designated as the primary column. This column will generally be the RTX-CLP, however, the analyst may specify either column as the primary column. The primary column must be used unless there is a documented reason (i.e. interference) for using the confirmation column for quantitation.
- 12.2.2.1 The identification and quantification of peaks is based on RT and the average calibration factor established during the initial calibration sequence.
- 12.2.2.2 A tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window. Each tentative identification must be confirmed by a second GC column of dissimilar stationary phase.
- 12.2.2.3 Quantitation of *multi-response analytes* can be done this way. The analyst may choose 4 to 6 of the largest peaks for quantitation. The peaks chosen for quantitation should have minimal co-elution with other peaks in the chromatogram. Use each peak chosen in the standard to calculate a calibration factor for that peak. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for the multi-response analyte in the sample.

13. QUALITY CONTROL

13.1 Method Blanks

13.1.1 Method blanks are an analyte-free matrix spiked with surrogate solution, extracted, and analyzed by the same procedure that is used with the associated samples.

13.1.1.1 In order to be acceptable, a method blank analysis cannot contain any of the analytes listed in this method above the reporting limit. All samples associated with a contaminated method blank must be re-extracted and reanalyzed unless the analyte concentration in the sample is greater than 20 times the amount found in the method blank, the analyte is not detected in the associated sample, or other approval is given directly by the supervisor.

13.1.2 The surrogate retention times must be within the retention time windows for both tetrachloro-m-xylene and decachlorobiphenyl. The surrogate recovery limits are established annually and distributed in the analytical laboratory. When surrogate recoveries in the method blank do not meet the recovery limits, the method blank is reanalyzed and/or the samples re-extracted.

13.2 Matrix Spike/Matrix Spike Duplicate

13.2.1 The analyte and surrogate retention times must be within the windows specified during the initial calibration or current continuing calibration.

13.2.2 The percent recovery and the relative percent difference between the recoveries of each of the compounds in the matrix spike samples will be calculated and reported by using the following equations:

$$\text{Matrix Spike Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

Where: SSR = Spike sample result
SR = Sample result
SA = Spike added

$$\text{RPD} = \frac{|\text{MSR} - \text{MSDR}|}{1/2 (\text{MSR} + \text{MSDR})} \times 100$$

Where: RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

13.2.3 The limits for matrix spike/matrix spike duplicate compound recoveries and RPD limits are established and distributed in the analytical area. Failure to meet these limits requires corrective action by the laboratory in accordance with S-GB-Q-027 *Corrective Action / Preventative Action Process*, most current revision or replacement.

13.3 Laboratory Control Spike

- 13.3.1 The percent recoveries and the relative percent difference between the recoveries of each of the compounds in the LCS/LCSD will be calculated and reported using the following equation:

$$\text{LCS/LCSD Recovery} = \frac{\text{LCSR}}{\text{SA}} \times 100$$

Where: LCSR = Lab control spike/duplicate recovery
SA = Spike added

$$\text{RPD} = \frac{|\text{LCSR} - \text{LCSDR}|}{1/2 (\text{LCSR} + \text{LCSDR})} \times 100$$

Where: RPD = Relative percent difference
LCSR = Laboratory control spike recovery
LCSDR = Laboratory control spike duplicate recovery

- 13.3.2 The limits for LCS/LCSD compound recoveries and RPD limits are established and distributed in the analytical area. Due to number of analytes fortified into the LCS, a small percentage of sporadic marginal failures may be tolerated (i.e. will not trigger re-extraction and analysis of the entire batch.) If more than 10% of the analytes are outside of the control limits, the samples are re-extracted or re-analyzed. For EPA 608 the limits must be met in EPA Method 608, Table 3.

13.4 Surrogate recoveries

- 13.4.1 Surrogate recoveries must be evaluated using laboratory established control limits. If both surrogate recoveries fail these criteria, re-extract the sample. One surrogate is allowed to be outside of the control limits.
- 13.4.2 Surrogates are not evaluated in samples where the surrogates are diluted below the low level standard concentration of the surrogates.
- 13.4.3 Surrogate recoveries will be calculated and reported using the following calculation:

$$\text{Surrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where: Q_d = Quantity determined by analysis
Q_a = Quantity added

14. DATA ANALYSIS AND CALCULATIONS

14.1 Calculations

Aqueous samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(C_f)(V_i)}$$

Soil, Solid, Biota samples:

$$\text{Concentration } (\mu\text{g/Kg}) \text{ (Dry weight basis)} = \frac{(A_x)(V_t)(D)}{(C_f)(W)(S)}$$

Where:

A_x	=	Area or peak height for an analyte
V_t	=	Final volume of extract in mL
D	=	Dilution factor
C_f	=	Average calibration factor
W	=	Initial sample weight (Kg)
V_i	=	Initial sample volume (L)
S	=	%Solids/100

Biota samples are typically reported on an “as is” or wet weight basis. The dry weight correction portion of the formula is typically not utilized.

14.2 Data Evaluation

14.2.1 For samples where a second analysis is performed at a dilution, the results that are reported from the second analysis are qualified with a “D”.

14.2.2 The analyst should compare the analyte concentrations between the two columns used for analysis. The RPD between the two columns should be < 40. If the RPD between the two results is greater than 40 but less than or equal to 100 the analyst should evaluate the chromatogram for interfering peaks that are co-eluting with the analyte. If an interference is determined to be present, report the lower of the two values and qualify the result with a “P” data qualifier. If no interference is determined to be present, report the higher of the two values and qualify the result with a “P” data qualifier.

14.2.3 Additional data qualifiers may be used dependent upon project specific requirement.

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1 See Section 13 and Table 7 Below:

Table 7: Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	SW846 8081A/B: Lab Generated Limits EPA 608: Attachment I, Method 608 Table 3, Range for Ps. Refer to the LIMS for acceptance limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	SW846 8081A/B: One MS/MSD set per preparation batch of up to 20 samples or one spiked sample per month. (EPA 608 one set for every 10 samples).	SW846 8081A/B: Lab Generated Limits EPA 608: Attachment I, Method 608 Table 3, Range for P. Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. <u>Exceptions:</u> 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample results are non-detect, the sample results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 See Section 13 and Table 7.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 See Section 13 and Table 7.

18. METHOD PERFORMANCE

- 18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.
 - 18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.
 - 18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*, most current revision or replacement. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.
 - 18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of the LOD and LOQ*, most current revision or replacement, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.
 - 18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, most current revision or replacement to demonstrate continuing competence. All results are stored in the QA office.

19. METHOD MODIFICATIONS

- 19.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 19.2 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- 19.3 Procedures identified as “Best Practices” by PACE 3P Programs will be incorporated into this document as minimum requirements for Pace Laboratories.
- 19.4 SW846 8081A and 8081B is a method written for aqueous and solid matrices, the laboratory has modified the method to accommodate biological tissues.
- 19.5 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

- 20.1 Any daily or periodic maintenance must be recorded in the instrument daily logbook. Additional information may be found in the most current revision of SOP: S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance*.

21. TROUBLESHOOTING

- 21.1 Please see the instrument operating manual for information on instrument troubleshooting.

22. SAFETY

- 22.1 **Standards and Reagents** - The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Standard solutions should be prepared in a hood.
- 22.2 **Samples** - Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.
- 22.3 A reference file of Safety Data Sheets (SDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

23. WASTE MANAGEMENT

- 23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Management and Handling*, most current revision or replacement.

24. POLLUTION PREVENTION

- 24.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.
- 24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. REFERENCES

- 25.1 Pace Quality Assurance Manual- most current version.
- 25.2 The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.
- 25.3 USEPA, SW-846, Method 8000C, Revision 3, March 2003.
- 25.4 USEPA, SW-846, Method 8081A, Revision 1, December 1996.
- 25.5 USEPA, SW-846, Method 8081B, Revision 2, February 2007
- 25.6 40CFR, Part 136, Appendix A, EPA Method 608.
- 25.7 S-GB-O-053 (most current revision or replacement), *Separatory Funnel Extraction by SW846 3510C*.
- 25.8 S-GB-O-054 (most current revision or replacement), *Ultrasonic Extraction by SW846 3550B*.
- 25.9 S-GB-O-031 (most current revision or replacement), *Extraction of Biological Samples for Organochlorine Pesticides/PCBs*.
- 25.10 S-GB-O-043 (most current revision or replacement), *Extraction of Toxaphene Using Automated Soxhlet*.

26. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA, ETC.

- 26.1 Attachment I: EPA Method 608, Table 3.

27. REVISIONS

SOP Number	Revision	Date
S-GB-O-027-Rev.03	Updated Signature Page. Section 9.3 & 9.4 – Update expiration dates. Section 9 – Table 1 – Updated with current information. Section 9 – Table 2 – Updated with current information. Section 9 – Table 3 – Updated with current information. Updated Section 10. Section 11.1.4 – Added PEM information. Section 11.4.3 – Deleted. Section 16 – Appendix A updated.	April 30, 2009
S-GB-O-027-Rev.05	Throughout Document: Updated Waste Handling and Management SOP to S-GB-S-006 Throughout Document: Removed all references to State of South Carolina criteria. Section 10.6.1 and 10.8.1: Added language of the acceptance criteria being the average of all compounds reported must be <15%. Section 11.2.3.2: Deleted Section 11.3 Calculations: Removed GPC From Calculation, it is a 1:1 dilution.	13Jul2011
S-GB-O-027-Rev.06	General: Updated SOP format Updated SOP references throughout document. Throughout Document: Added language to address update to MUR and SW846 8081B..	13Jan2014
S-GB-O-027-Rev.07	Table 2: Included TCLP/SPLP Leach hold-times. Table 3: Updated to include 40GCSG. Table(s) 4 and 5: Updated to current standard amounts and concentrations. Section 11.6.1: Clarified second source requirement. Section 11.8.3: Added additional CCV requirement. Section 25: Included Pace and TNI references.	10Dec2014
S-GB-O-027-Rev.08	Title Page, Section 1.1 and 25: Added EPA 608 References. Section(s) 11.3, 11.5, 11.6, 11.7: Added EPA 608 Method requirements. Section 12.1.1: Added MS/MSD per every 10 samples. Table 7: Added. Section 13.2.3: Updated SOP reference. Attachment I: EPA 608, Table 3 added..	17Jun2015

Attachment I: Method 608 Table 3

Table 3—QC Acceptance Criteria—Method 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for \bar{X} (µg/L)	Range for P, P _s (%)
Aldrin	2.0	0.42	1.08 - 2.24	42 - 122
α-BHC	2.0	0.48	0.98 - 2.44	37 - 134
β-BHC	2.0	0.64	0.78 - 2.60	17 - 147
δ-BHC	2.0	0.72	1.01 - 2.37	19 - 140
γ-BHC	2.0	0.46	0.86 - 2.32	32 - 127
Chlordane	50	10.0	27.6 - 54.3	45 - 119
4,4'-DDD	10	2.8	4.8 - 12.6	31 - 141
4,4'-DDE	2.0	0.55	1.08 - 2.60	30 - 145
4,4'-DDT	10	3.6	4.6 - 13.7	25 - 160
Dieldrin	2.0	0.76	1.15 - 2.49	36 - 146
Endosulfan I	2.0	0.49	1.14 - 2.82	45 - 153
Endosulfan II	10	6.1	2.2 - 17.1	D - 202
Endosulfan Sulfate	10	2.7	3.8 - 13.2	26 - 144
Endrin	10	3.7	5.1 - 12.6	30 - 147
Heptachlor	2.0	0.40	0.86 - 2.00	34 - 111
Heptachlor epoxide	2.0	0.41	1.13 - 2.63	37 - 142
Toxaphene	50.0	12.7	27.8 - 55.6	41 - 126
PCB-1016	50	10.0	30.5 - 51.5	50 - 114
PCB-1221	50	24.4	22.1 - 75.2	15 - 178
PCB-1232	50	17.9	14.0 - 98.5	10 - 215
PCB-1242	50	12.2	24.8 - 69.6	39 - 150
PCB-1248	50	15.9	29.0 - 70.2	38 - 158
PCB-1254	50	13.8	22.2 - 57.9	29 - 131
PCB-1260	50	10.4	18.7 - 54.9	8 - 127

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).

\bar{X} = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).

P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).

D = Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.


STANDARD OPERATING PROCEDURE

Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by SW846-8082A and EPA Method 608

Reference Methods: SW-846 Method 8082A / EPA 608

SOP NUMBER:	S-GB-O-047-REV.05
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-047-REV.04

APPROVAL

	07/02/15
Nils Melberg, Laboratory General Manager	Date

	6/18/15
Kate Grams, Laboratory Quality Manager	Date

	6/18/15
Chris Haase, Department Manager	Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to determine the concentration of PCBs in water, soil, sediment, waste, and biological samples in accordance with SW846 Method 8082A / EPA 608. Samples for analysis are prepared by SW846 Method 3510C, 3540C, 3541, and 3580A. See Section 25 for list of reference SOPs.

2. SUMMARY OF METHOD

- 2.1 Sample extracts are prepared for analysis by the appropriate sample preparation method. The procedures for extract preparation are described in separate SOPs. A volume of sample extract is injected into a GC and compounds in the effluent are detected by an ECD based on an operating program set up to achieve optimum separation and quantitation of target analytes.
- 2.2 Retention time windows, in combination with characteristic elution patterns from a dual-column analysis, are used in the identification of PCBs as Aroclors.
- 2.3 PCBs are quantified as Aroclor mixtures by comparison of their ECD response on a single column with a calibration curve(s) constructed from the response(s) of authentic standards.
- 2.4 Results are reported in parts per billion ($\mu\text{g/kg}$ or $\mu\text{g/L}$). Soil and sediment sample results are corrected for moisture and reported on a dry weight basis. Biological results are reported based on wet weight, or “as is” basis

3. SCOPE AND APPLICATION

- 3.1 **Personnel:** This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/electron capture detection (GC/ECD) systems and interpretation of complex chromatograms. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.
- 3.2 **Parameters:** A list of the Aroclors routinely analyzed, their CAS numbers and Pace Reporting Levels (PRLs) are shown in Section 5, Table 1. PRLs are subject to change based on current analytical system performance and actual sample matrices.

4. APPLICABLE MATRICIES

- 4.1 This method is used to determine the concentration of PCBs in extracts prepared from water, soil, sediment, waste, and biological samples.

5. LIMITS OF DETECTION AND QUANTITATION

- 5.1 The reporting limit (PQL) of this method for Polychlorinated Biphenyls is listed in Table 1 below. All current MDLs are listed in the LIMs and are available by request from the Quality Department.

Table 1: Analyte List and Reporting Limits

Aroclor	CAS #	Aqueous PQL (µg/L)	Solid PQL (µg/Kg)	Biota PQL (µg/Kg)
AR1016	12674-11-2	0.5	50	25
AR1221	11104-28-2	0.5	50	25
AR1232	11141-16-5	0.5	50	25
AR1242	53469-21-9	0.5	50	25
AR1248	12672-29-6	0.5	50	25
AR1254	11097-69-1	0.5	50	25
AR1260	11096-82-5	0.5	50	25
AR1262*	37324-23-5	0.5	50	25
AR1268*	11100-14-4	0.5	50	25
Total PCB	NA	0.5	50	25

***Note: Aroclor 1262 and 1268 only analyzed per client request**

6. INTERFERENCES

- 6.1 Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that leads to discrete artifacts and/or elevated baselines. Phthalate esters are common contaminants that result from contact with flexible plastics. Contact with common plastics or rubber products must be avoided. Lab ware should be constructed of glass, stainless steel, or PTFE, must be thoroughly cleaned and dried prior to use, and should be rinsed with the appropriate solvent immediately before use.
- 6.2 Elemental sulfur is a common environmental contaminant in many soil, sediment and leachate samples, producing a broad peak that will confound analysis of early eluting analytes. Sulfur may be removed from extracts by treatment with copper granules or similar procedure described in a separate SOP.
- 6.3 Waxes, lipids, and other similar high molecular weight materials may be co-extracted from samples typically resulting in baseline elevation during GC analysis. These interferences may be removed by sulfuric acid clean up and/or column chromatography cleanup using Florisil or gel permeation chromatography (GPC), all of which are described in separate SOPs. Other halogenated pesticides and similar industrial chemicals, which can interfere with analytes of interest, may be removed by these procedures as well.
- 6.4 All solvents, reagents, glassware, and sample processing hardware must be routinely demonstrated to be free from interferences under the conditions of the analysis by monitoring method blanks and taking corrective action as required.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1 **General Procedures** – Procedures for sample collection, preservation, and handling are described in the separate sample preparation SOPs.

Table 2: Sample Collection, Preservation, Storage and Hold time

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One 1L amber glass	None	≤6°C	365 days
Soil/Solid (non-aqueous)	One 8oz wide glass jar	None	≤6°C	365 days
Biological Tissue	--	None	≤ -10°C	365 days or longer per client request prior to extraction, typical extraction hold time do not apply.
Extracts	ONE 10mL glass vial or 5mL copper clean vial.	None	≤6°C	365 days

8. DEFINITIONS

8.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

8.2 **Extract** – A solution of contaminants extracted and concentrated from a sample.

9. EQUIPMENT AND SUPPLIES

9.1 Instrumentation

9.1.1 **Gas Chromatograph (GC)** – Hewlett Packard (HP) 5890 equipped with dual ECDs or HP 6890 equipped with dual μ ECDs.

9.1.2 **GC Autosampler** – HP 7673A (5890) or HP 7863 (6890).

9.1.3 **GC Columns** – Two of the following capillary columns may be used:

9.1.3.1 RTX CLPesticides I, 30m x 0.32mm I.D. (Restek)

9.1.3.2 RTX CLPesticides II, 30m x 0.32mm I.D. (Restek)

9.1.3.3 DB-1701, 30m x 0.32mm I.D. (J&W Scientific)

9.1.3.4 DB-5, 30m x 0.32mm I.D. (J&W Scientific)

9.1.4 **Data Processor** – TurboChrom IV or HP ChemStation.

9.1.5 **Printer** – HP LaserJet 5Si or equivalent.

9.2 Glassware and Materials

- 9.2.1 Gastight Syringes – any size ranging from 10µL to 1000µL (Hamilton series 1000 or equivalent).
- 9.2.2 Autosampler Vials – 2.0mL glass vials with crimp top caps.
- 9.2.3 Helium Gas – Airgas Ultra high purity or equivalent
- 9.2.4 Nitrogen Gas – Airgas Ultra high purity or equivalent

10. REAGENTS AND STANDARDS

- 10.1 **Solvents** – Hexane and acetone, pesticide grade. All solvents are stored at room temperature and environmental conditions.

Table 3: Solvents

Reagent	Purity	Manufacturer	Vendor	Catalog #	Expiration Date
Hexane	NS Grade	Burdick & Jackson	MG Scientific	B&J-217-4	Manufacturer Exp date or 2 year from receipt
Acetone	Pesticide Grade	Burdick & Jackson	MG Scientific	B&J-010-4	Manufacturer Exp date or 2 year from receipt

- 10.2 **Analytical Standards** – Prepared from stock standard solutions and are required for initial calibration and continuing calibration checks (Table 4). The following describes the contents of each type of solution:
 - 10.2.1 **Calibration and Calibration Check Standards** – Five concentration levels of calibration solutions are prepared containing equal amounts of Aroclors 1016 and 1260 (combined in the same solution named AR1660 throughout this document), as well as the surrogates decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TCMX). A single point calibration standard is required for the other Aroclor mixtures preferably at the mid-point level of the AR1016/1260 curve. A calibration check solution (ICV) is also prepared at the mid-level concentration of AR1016/1260 from second source materials.
 - 10.2.2 **Surrogate Standard Spiking Solution** – contains decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TCMX) and is spiked into all samples prior to extraction.
 - 10.2.3 **Matrix Spiking Solutions** – contain an Aroclor mixture that is spiked into all appropriate QC samples (LCS, MS, and MSD) prior to extraction. The Aroclor(s) spiked and/or spike amounts may be adjusted when prior knowledge of the type or concentration of Aroclor(s) present in the sample matrix is known, or to comply with project requirements.

Table 4: Standard Stock Solutions

Standard	Concentration	Manufacturer	Catalog #	Expiration Date
Pesticide Surrogate Mix	200µg/mL each in Acetone	Restek Corporation or equivalent	32000	Manufacturer's recommended expiration date for unopened ampulated standards. 1 year after ampule is opened or on expiration date, whichever is sooner.
Aroclor 1016 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32006	
Aroclor 1221 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32007	
Aroclor 1232 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32008	
Aroclor 1242 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32009	
Aroclor 1248 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32010	
Aroclor 1254 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32011	
Aroclor 1260 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32012	
Aroclor 1262 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32409	
Aroclor 1268 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32410	
Aroclor 1016	1000µg/mL in Isooctane	Supelco or equivalent	4-8097	
Aroclor 1260	1000µg/mL in Isooctane	Supelco or equivalent	4-4809	

***Note: Aroclor 1262 and 1268 only analyzed upon client request**

10.3 Preparation of Analytical Standard Solutions – Standards are prepared from commercially available stock solutions. The sources of the stock solutions, recipes for preparing dilutions and working standards, and concentrations in all solutions are shown in Table 5. All standards are prepared in hexane and stored in amber vials with PTFE-lined screw caps at ≤6 °C.

10.4 Stability of Analytical Standards – Stock solutions of Aroclor mixtures must be replaced within 1 year of preparation. All dilutions and working standard solutions must be replaced within 6 months of preparation or sooner if the standards show signs of degradation. As each standard from the vendor is opened, record all pertinent information in the stock standard logbook. Record all standard preparations in the working standard logbook.

Table 5: Preparation of Analytical Standard Solutions.

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
TCMX/DCB Stock Solution	Pesticide Surrogate Mix	1000µL	20mL of Hexane	10µg/mL	1 year from date of preparation or the expiration date listed for the stock source, whichever is sooner.
AR1221 Stock Solution	Aroclor 1221 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1232 Stock Solution	Aroclor 1232 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1242 Stock Solution	Aroclor 1242 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1248 Stock Solution	Aroclor 1248 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1254 Stock Solution	Aroclor 1254 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1262 Stock Solution	Aroclor 1262 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1268 Stock Solution	Aroclor 1268 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1660 Stock Solution	Aroclor 1016 Mix Aroclor 1260 Mix	1000µL each	10mL of Hexane	100µg/mL each	
AR1660 ICV Stock Solution	Aroclor 1016 Aroclor 1260	1000µL each	10mL of Hexane	100µg/mL each	
AR1221-3 Calibration Standard	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 500µL TCMX/DCB 500µL	100mL of Hexane	AR1221 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. From date of preparation or the expiration date listed for the stock source, whichever is sooner.
AR1232-3 Calibration Standard	AR1232 Stock Solution TCMX/DCB Stock Solution	AR1232 500µL TCMX/DCB 500µL	100mL of Hexane	AR1232 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1242-3 Calibration Standard	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 500µL TCMX/DCB 500µL	100mL of Hexane	AR1242 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1248-3 Calibration Standard	AR1248 Stock Solution TCMX/DCB Stock Solution	AR1248 500µL TCMX/DCB 500µL	100mL of Hexane	AR1248 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1254-3 Calibration Standard	AR1254 Stock Solution TCMX/DCB Stock Solution	AR1254 500µL TCMX/DCB 500µL	100mL of Hexane	AR1254 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1262-3 Calibration Standard	AR1262 Stock Solution TCMX/DCB Stock Solution	AR1262 500µL TCMX/DCB 500µL	100mL of Hexane	AR1262 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1268-3 Calibration Standard	AR1268 Stock Solution TCMX/DCB Stock Solution	AR1268 500µL TCMX/DCB 500µL	100mL of Hexane	AR1268 0.5µg/mL TCMX/DCB 0.05µg/mL	

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1660-1 Calibration Standard and PRLS	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 50µL TCMX/DCB 100µL	100mL of Hexane	AR1660 0.05µg/mL TCMX/DCB 0.01µg/mL	6 mo. From date of preparation or the expiration date listed for the stock source, whichever is sooner.
AR1660-2 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 200µL TCMX/DCB 200µL	100mL of Hexane	AR1660 0.2µg/mL TCMX/DCB 0.02µg/mL	
AR1660-3 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 1000µL TCMX/DCB 1000µL	200mL of Hexane	AR1660 0.5µg/mL TCMX/DCB 0.05µg/mL	
AR1660-4 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1660 0.8µg/mL TCMX/DCB 0.10µg/mL	
AR1660-5 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1660 1.0µg/mL TCMX/DCB 0.15µg/mL	
AR1660-3 ICV Calibration Standard	AR1660 ICV Stock Solution TCMX/DCB Stock Solution	AR1660 500µL TCMX/DCB 500µL	100mL of Hexane	AR1660 0.5µg/mL TCMX/DCB 0.05µg/mL	

***Note: Aroclor 1262 and 1268 only analyzed upon client request**

11. CALIBRATION AND STANDARDIZATION

11.1 Initial Calibration (ICAL)

11.1.1 Analysis of Standards

- 11.1.1.1 The initial calibration includes analysis of a five-point calibration curve of AR1660 at concentrations of 0.05, 0.2, 0.5, 0.8, and 1.0µg/mL, which includes TCMX and DCB at concentrations of 0.01, 0.02, 0.05, 0.1, and 0.15µg/mL respectively. Inject a single point standard of Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268 at 0.5µg/mL.
- 11.1.1.2 Other calibration ranges may be substituted to meet expected concentrations of samples to be analyzed. If historical data indicates a specific Aroclor is present, a five point initial calibration may be performed for the Aroclor of concern instead of using the AR1660 mixture.
- 11.1.1.3 Three to ten (preferably seven) peaks must be selected for each Aroclor, except for Aroclor 1221 which only requires a minimum of three peaks. The peaks chosen for quantitation should be at least 25% of the height of the largest peak in each Aroclor and should have minimal co-elution with the peaks of other Aroclors.

11.1.2 Retention Time (RT) – Retention time windows are used for compound identifications in samples. The RT for all components in all standards must be within the windows specified for both columns.

- 11.1.2.1 Make at least three injections of all analytes of interest over a 72-hour period.
- 11.1.2.2 Record the retention time for each selected peak for each Aroclor mixture, to three decimal places. Calculate the mean and standard deviation for each peak.
- 11.1.2.3 The width of the retention time window is defined as ± 3 standard deviations of the mean established. The minimum retention window will be ± 0.03 minutes.
- 11.1.2.4 Establish the center of the RT window for each Aroclor mixture and surrogate using the absolute RT from the calibration verification standard at the beginning of the analytical shift. Optionally, the Initial Calibration RT windows may continue to be used as long as method criteria are met. For samples run during the same shift as an initial calibration, use the RT of the mid-point standard in the Initial calibration as the center of the RT window.

11.1.2.5 When conducting Aroclor analysis, it is important to determine that common single-component pesticides such as DDT, DDD, and DDE do not elute at the same retention times as the target Aroclors. In conjunction with determining the retention time windows, the analyst should analyze a standard containing the DDT analogs. The standard only needs to be analyzed when the retention time windows are being determined. It is not part of the routine initial calibration or calibration verification steps in the method, nor are there any performance criteria with the analysis of the standard. If it is determined that any of the DDT analogs elute at the same retention time as an Aroclor peak that was chosen for quantitation, then the analyst must either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Aroclor and does not correspond to a peak from a DDT analog.

11.1.3 **Response Factors (RF)** – Individually tabulate the area responses for each of the five or more peaks selected for each Aroclor versus concentration of the five-point calibration standards for each GC column. Calculate RF for each peak using the following equation:

$$RF = \frac{A_x}{C_x}$$

Where:

A_x = Total area of analyte response.

C_x = Concentration of the analyte in the solution (µg/mL).

11.1.4 **Acceptance Criteria** –

11.1.4.1 SW-846 8082A: The percent relative standard deviation (%RSD) of the five calibration factors for each peak of each Aroclor, (1016 and 1260) along with the surrogates must be ≤ 20%. If this is the case, linearity can be assumed, and the average RF can be used for quantitation. If the %RSD is >20%, a linear calibration curve may be used if the correlation coefficient is ≥ 0.99. **The results for both columns must meet calibration acceptance criteria.**

11.1.4.2 EPA Method 608: The percent relative standard deviation (%RSD) of the five calibration factors for each peak of each Aroclor, (1016 and 1260) along with the surrogates must be ≤ 10%. If this is the case, linearity can be assumed, and the average RF can be used for quantitation. If the %RSD is >10%, a linear calibration curve may be used if the correlation coefficient is ≥ 0.99. **The results for both columns must meet calibration acceptance criteria.**

11.1.5 **Initial Calibration Verification (ICV)** – In order to consider the initial calibration acceptable, an ICV standard must be analyzed. The ICV standard must be from a second source stock and meet the same criteria as the continuing calibration verification standard before the initial calibration may be considered valid.

11.1.6 **Continuing Calibration Verification (CCV)** – A midpoint calibration check standard must be injected at the beginning and end of each 12-hour analysis period, and at intervals of not less than once every 20 samples, for calibration verification

11.1.7 **Acceptance Criteria** –

11.1.7.1 SW-846 8082A: The percent difference (%D) is determined for every analyte and must be within $\pm 20\%$ of the calibration curve. Calculate %D for each peak using the following equation:

$$\%D = \left(\frac{R_1 - R_2}{R_1} \right) \times 100$$

Where:

R_1 = Mean Response factor from the ICAL

R_2 = RF calculated from the CCV

11.1.7.1.1 First determine whether the average %D for all of the peaks for each specific Aroclor with a five-point calibration is $\leq 20\%$. Each individual Aroclor must be evaluated separately. For example, the average %D for all of the peaks used for quantitation of AR1016 must be $\leq 20\%$. If the Aroclors themselves are acceptable, evaluate the %D for each surrogate. If the %D is $\leq 20\%$ for each individual Aroclor and surrogate, the continuing meets the acceptance criteria.

11.1.7.1.2 If the ending calibration verification standard exceeds 20%D criteria on the high side (i.e., an increase in sensitivity) samples that had no Aroclors detected do not need to be reanalyzed. If the continuing calibration standard criterion is exceeded on the low side (i.e. a drop in sensitivity) all samples analyzed since the last acceptable CCV must be re-analyzed.

11.1.7.2 EPA Method 608: The percent recovery is determined for every analyte and must be within $\pm 15\%$ of the predicted response. Calculate percent recovery for each peak using the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

11.1.7.2.1 First determine whether the % recovery for all of the peaks for each specific Aroclor with a five-point calibration is within 15% of the predicted response. If the Aroclors themselves are acceptable, evaluate each surrogate to determine if their recovery is within 15% of the predicted response.

11.1.7.2.2 If the ending calibration verification standard exceeds the 15% recovery criteria on the high side (i.e., an increase in sensitivity) samples that had no Aroclors detected do not need to be reanalyzed. If the continuing calibration standard criterion is exceeded on the low side (i.e. a drop in sensitivity) all samples analyzed since the last acceptable CCV must be re-analyzed

11.1.8 **All samples must be bracketed by acceptable calibration verifications on both columns.** Perform corrective action such as injection port or column maintenance. Prior to the analysis of any subsequent samples acceptable calibration verification must be established. In the event that this cannot be achieved, a new initial calibration must be performed.

11.1.9 **Reporting Limit Verification Standard (RLVS)** – For every five point Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed. The RLVS is analyzed prior to any samples being analyzed, and monthly thereafter. The limits are +/- 40% of the true concentration. The analysis of this standard demonstrates the instruments ability to report down to the reporting limit with known accuracy. If outside the limits, reevaluate the low level standards. If still outside the limits, recalibrate.

12. PROCEDURE

12.1 **Sample Preparation** – All sample extracts and standard solutions must be allowed to warm to room temperature before analysis.

12.2 **GC/ECD System Preparation** – Verify instrument parameters as set up for current operating conditions.

12.2.1 GC Column Conditions

Carrier Gas	UHP Helium
Flow Rate	3.4 mL/min.
Make-up Gas	UHP Nitrogen
Flow Rate	35.0 mL/min.
Detector Temp.	300°C
Injector Temp.	205°C
Injection	Splitless

12.2.2 GC Temperature Program

Initial Temp.	110°C
Initial Time	1.5 min.
Rate 1	20.00°C/min.
Final Temp. 1	140°C
Final Time 1	0.00 min.
Rate 2	11.00°C/min.
Final Temp. 2	280°C
Final Time 2	5.00 min.
Rate 3	20.00°C/min.
Final Temp. 3	300°C
Final Time 3	3.00 min.

12.3 **Batch Sequence** – Generate a sequence to run a batch of samples and the associated quality control samples.

12.3.1 **Initial Calibration** – For example, the batch for initial calibration should include the following:

Series of 2-3 Primes
Solvent Blank (Hexane)
AR1660-1 (0.05µg/mL)
AR1660-2 (0.2µg/mL)
AR1660-3 (0.5µg/mL)
AR1660-4 (0.8µg/mL)
AR1660-5 (1.0µg/mL)
Solvent Blank (Hexane)
AR1660-3 ICV
AR1660-1 (0.1µg/mL) (PRLS)

12.3.2 **Sample Analysis** – For example, the typical batch for analysis of PCBs should include the following:

AR1660-301 CCV (0.5µg/mL)
(20 samples or 12-hour period)
Method Blank
Laboratory Control Spike
Samples
Matrix Spike/Matrix Spike Duplicate
Duplicate Sample(s)
AR1660-302 CCV (0.5µg/mL)

12.4 **Load Autosampler** – Load the autosampler with the appropriate primes, solvent blanks, standards and samples for the batch as it was created.

12.5 **Analyze Samples** – Analyze all standards, quality control samples, and environmental samples.

12.5.1 The method blank and LCS extracted along with the samples should be analyzed on the same instrument as the samples.

- 12.5.2 If the analyst determines that interferences could be removed by sulfuric acid cleanup and/or sulfur removal, then the analyst will perform the necessary cleanups and re-analyze the samples. The blank and LCS will also undergo the same cleanups and be re-analyzed.

12.6 Qualitative Analysis of Results

12.6.1 Identification

- 12.6.1.1 To be identified as an Aroclor, peaks present in a sample extract must fall within the established retention time window for a specific Aroclor. Once the Aroclor pattern has been tentatively identified, compare the responses of 3 to 10 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. Overlay comparison of sample chromatograms with standard chromatograms may be required to clearly identify patterns.
- 12.6.1.2 Since the chromatograms for many Aroclor mixtures overlap, the presence of multiple mixtures may complicate their quantitation. Also, environmental “weathering” of PCBs may complicate reliable identification and quantitation.

12.6.2 Confirmation

- 12.6.2.1 Confirmation is generally required using a second GC column of dissimilar stationary phase. When dual-column analysis is performed for confirmation, the same initial and continuing calibration criteria apply to both columns.
- 12.6.2.2 Since Aroclors provide distinct multiple peak patterns which may be identified by an experienced analyst, confirmation on the second column may be based upon pattern recognition.

13. QUALITY CONTROL

13.1 Calibration Checks

- 13.1.1 **ICAL** – If initial calibration criteria are not met, check standards preparation procedure for errors. Prepare new standards as required and re-run the calibration.
- 13.1.2 **Continuing Calibration Verification** – If the CCV criteria are not met, check system parameters, identify and correct likely causes, and re-run the check. An acceptable check is required to report sample results for the applicable batch.
- 13.1.3 **Reporting Limit Verification Standard (RLVS)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate. The AR1660 0.1µg/mL initial calibration standard is used as the PRLS

- 13.2 **Surrogate Recoveries** – Surrogate compound(s) must be added to all samples, spikes, control samples and method blanks, prior to analysis as indicators of method accuracy. Laboratory-based accuracy limits should be used for acceptance criteria. For Biota samples, if laboratory limits chart to narrow, limits will be set to 60-130%. If these criteria are not met, check system parameters, identify and correct likely causes, and re-run the samples.
- 13.2.1 If **both** surrogate recoveries fail this criterion, re-extraction of the sample may be necessary. If surrogate recoveries are higher than the acceptance criteria and target compounds are less than the reporting limit, the results may be reported with an appropriate footnote. If recoveries appear out of control due to sample matrix, report the results with an appropriate footnote.
- 13.2.2 One surrogate is allowed to be outside of the control limits. For instance, if an interfering peak obscures one surrogate, then that one surrogate may be excluded. The surrogate is considered diluted out and not evaluated when the dilution performed brings the theoretical on-column concentration below the concentration of the low standard in the initial calibration curve.
- 13.2.3 For samples run from the state of South Carolina, both surrogates must recover between 70-130%.
- 13.3 **Method Blank** –The method blank must not contain analyte responses at or above the reporting limit. If the results are not acceptable, re-analyze the method blank. If the problem persists, conduct maintenance to clean the analytical system. An acceptable method blank is required to report sample results for the applicable batch.
- 13.3.1 One surrogate is allowed to be outside of the control limits. For instance, if an interfering peak obscures one surrogate, then that one surrogate may be excluded. The surrogate is considered diluted out and not evaluated when the dilution performed brings the theoretical on-column concentration below the concentration of the low standard in the initial calibration curve.
- 13.3.2 If the blank contains any analyte of interest above the reporting limit, all of the associated samples, matrix spikes, and laboratory control spikes **must** be re-extracted unless the sample concentration is greater than 20X the amount found in the blank or the analyte is not detected in an associated sample. For Wisconsin projects this criteria will be “Above the LOD”.
- 13.4 **LCS Recoveries** – One LCS must be analyzed with each batch of 20 samples. Laboratory-based accuracy limits should be used to for acceptance criteria. For EPA Method 608 see Table 9 for acceptance criteria. For Biota samples, if laboratory limits chart to narrow, limits will be set to 60-130%. An acceptable LCS is required to report sample results for the applicable batch.
- 13.4.1 If the laboratory control spike does not meet the recovery criteria, the results of all QC performed with the batch will be evaluated by the analyst. Corrective actions include re-extraction of the samples or reanalysis of the extracts.
- 13.4.2 For samples run from the state of South Carolina, Ar1016 and 1260 must recover between 70-130%.

- 13.5 One LCSD must be analyzed with each batch of 20 samples if inadequate sample is available to perform a MS/MSD. Laboratory-based accuracy limits should be used to for acceptance criteria. An acceptable LCSD is required to report sample results for the applicable batch.

13.6 **MS/MSD Recoveries –**

- 13.6.1 SW-846 8082A: One MS/MSD pair should be analyzed with each batch of 20 samples. Laboratory-based accuracy limits should be used to for acceptance criteria. The sample use for the MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows.
- 13.6.2 EPA Method 608: One MS/MSD pair should be analyzed at a rate of 10% of the samples being analyzed. See Table 9 from Method 608 for acceptance criteria. The sample used for the MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows.
- 13.6.3 If a matrix spike recovery fails this criterion, the recovery of the other spiked sample in the MS/MSD pair should be evaluated. If recovery failures are duplicated then the sample matrix is suspected as the problem and the data should be flagged and the failures discussed in the sample narrative.

- 13.7 **Duplicate and MS/MSD RPDs** – Five percent of all environmental samples should be analyzed in duplicate. A MS/MSD pair is also an acceptable duplicate analysis. If results are not acceptable, check for possible sample preparation problems and re-analyze if needed. Report the results with an appropriate data qualifier.

14. DATA ANALYSIS AND CALCULATIONS

14.1 Calculate Results

- 14.1.1 The amount of Aroclor is calculated using the individual response factor (single point) for each of the 3 -10 characteristic peaks chosen for quantitation of that specific Aroclor. If Aroclor 1016 and/or 1260 is being quantified use the average response factor from the AR1660 curve. Use the single point response factor from the initial calibration for all other Aroclors. Surrogates are quantified based on the average response factors for TCMX and DCB analyzed with the AR1660 curve. A concentration is determined using each of the characteristic peaks and then those concentrations are averaged to determine the on-column concentration of that Aroclor.
- 14.1.2 If the initial on-column result of a sample extract exceeds the calibration range, the extract must be diluted and re-analyzed. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve. The GC data system will calculate concentration of each parameter as µg/mL on-column in the extract. Concentrations in samples are then calculated based on sample size, total volume of the final extract, any dilution factor, and any correction factor.

14.1.2.1 Water and Water-Miscible Waste Samples

$$\text{Final Concentration } (\mu\text{g/L}) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(V_o)}$$

Where:

C_x = On-column concentration in extract ($\mu\text{g/mL}$).

DF = Dilution factor.

U_f = Correction factor.

V_t = Volume of final extract (μL).

V_i = Volume injected (μL).

V_o = Volume of water sample extracted (mL).

14.1.2.2 Soil/Solid, Waste and Biological Samples

$$\text{Final Concentration } (\mu\text{g/Kg}) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(W_s)(S)}$$

Where:

C_x = On-column concentration in extract ($\mu\text{g/mL}$).

DF = Dilution factor.

U_f = Correction factor.

V_t = Volume of final extract (μL).

V_i = Volume injected (μL).

W_s = Weight of sample extracted (g).

S = Percent Solids (biological samples not corrected for percent solids).

14.1.3 Air monitoring sample result calculations use a time/volume relationship to calculate PCB concentration. (See Attachment I for example.)

$$\text{Final Concentration } (\mu\text{g/L}) \text{ or } (\text{mg/m}^3) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(V_s)}$$

Where:

C_x = On-column concentration in extract ($\mu\text{g/mL}$).

DF = Dilution factor.

U_f = Correction factor.

V_t = Volume of final extract (μL).

V_i = Volume injected (μL).

V_s = Volume of air sampled (L).

S = Percent Solids (biological samples not corrected for percent solids).

Volume of air sampled L = LPM * T

Where:

LPM = Liters per Minute of air sampled.

T = Sampling time in minutes.

14.2 **Quality Control Results** – Calculate recoveries for the surrogates in all samples; spiked analytes in LCS and MS/MSD samples; and Relative Percent Differences (RPD) for duplicate and MS/MSD samples.

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Table 6: Quality Control

Analytical Method ⇨ Quality Control Measure ⇩	SW846 8082A and EPA 608
Initial Calibration	Minimum of five levels; lowest level at or below PQL.
Initial Calibration Verification Standard (ICV)	After every initial calibration.
Calibration Verification Standard (CCV)	One at the beginning of a 12 hour time clock, every 20 injections or more frequent.
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	SW-846 8082A: One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. EPA 608: One pair per 10 samples analyzed.
Method Validation	Annually
MDL	Annually
Surrogate Standards	Added to every sample.
Reporting Limit Verification Standard (RLVS)	After every calibration and monthly thereafter.

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Table 7: Corrective Actions

Analytical Method Acceptance Criteria⇒ Calibration Measure ↓	SW846 8082A / EPA 608 Frequency	Acceptance Criteria
Initial Calibration	<ul style="list-style-type: none"> • Installation of new column • Creation of new analytical method • CCV fails criteria 	<ul style="list-style-type: none"> • Use Average Calibration and relative standard deviation (%RSD) is ≤ 20%.
Initial Calibration Verification (ICV)	<ul style="list-style-type: none"> • Immediately after the ICAL. 	<ul style="list-style-type: none"> • Percent Difference (%D) is ≤ 20%
Continuing Calibration Verification (CCV)	<ul style="list-style-type: none"> • At the beginning of every 12 hour shift, every 20 injections, or more frequent 	<ul style="list-style-type: none"> • Percent Difference (%D) is ≤ 20%
Laboratory Control Spike and Duplicate	<ul style="list-style-type: none"> • One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. 	<ul style="list-style-type: none"> • In house limits determined.
Matrix Spike and Duplicate	<ul style="list-style-type: none"> • One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. 	<ul style="list-style-type: none"> • In house limits determined.
MDL's	<ul style="list-style-type: none"> • Annually 	<ul style="list-style-type: none"> • 1-10 times the MDL should be equal to the spike concentration that was used to determine the MDL's
Method Validation	<ul style="list-style-type: none"> • Annually 	<ul style="list-style-type: none"> • In house limits determined.
Surrogate Standards	<ul style="list-style-type: none"> • Added to every sample. 	<ul style="list-style-type: none"> • In house limits determined.
Reporting Limit Verification Standard (RLVS)	<ul style="list-style-type: none"> • After every initial calibration and monthly thereafter 	<ul style="list-style-type: none"> • 60-140%

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 See Section 13, 15 and 16

18. METHOD PERFORMANCE

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*, most current revision or replacement. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

- 18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of the LOD and LOQ*, most current revision or replacement, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.
- 18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, most current revision or replacement, to demonstrate continuing competence. All results are stored in the QA office.

19. METHOD MODIFICATIONS

- 19.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 19.2 All Major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- 19.3 Procedures identified as “Best Practices” by PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- 19.4 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.
- 19.5 Calibration standards are to be made in Hexane, not in iso-octane as stated in EPA method 608.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

- 20.1 Any daily or periodic maintenance must be recorded in the instrument daily logbook. Additional information may be found in the most current revision of SOP: S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance*.

21. TROUBLESHOOTING

- 21.1 Please see the instrument operating manual for information on instrument troubleshooting.

22. SAFETY

- 22.1 The toxicity, or carcinogenicity, of many chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Reduce exposure by the use of hood, gloves, lab coats and safety glasses.
- 22.2 A reference file of Safety Data Sheets (SDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

23. WASTE MANAGEMENT

- 23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management*.

24. POLLUTION PREVENTION

- 24.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.
- 24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. REFERENCES

- 25.1 Pace Quality Assurance Manual- most current version.
- 25.2 The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.
- 25.3 USEPA, SW-846, Method 8082A, “Polychlorinated Biphenyls (PCBs) by Gas Chromatography”, February 2007.
- 25.4 USEPA, SW-846, Method 8000B, “Determinative Chromatographic Separations”, December 1996.
- 25.5 Appendix A to part 136, Methods for organic chemical analysis of municipal and industrial wastewater, “Method 608 – Organochlorine pesticides and PCBs”
- 25.6 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-053, “*Separatory Funnel Extraction*”, most current revision or replacement.
- 25.7 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-031, “*Extraction of Biological Samples for Organochlorine Pesticides/PCBs*”, most current revision or replacement.
- 25.8 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-040, “*Extraction of Wipes and Oil for PCB Analysis*”, most current revision or replacement.
- 25.9 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-041, “*Extraction of PCBs Using the Automated Soxhlet*”, most current revision or replacement.
- 25.10 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-032, “*Gel Permeation Chromatography*”, most current revision or replacement.
- 25.11 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-034 *Sulfuric Acid Cleanup*”, most current revision or replacement.
- 25.12 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-036, “*Florisil Cleanup for PCBs*”, most current revision or replacement.
- 25.13 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-038, “*Silica Gel Cleanup of Organochlorine Pesticides and PCBs*”, most current revision or replacement.
- 25.14 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-039, “*Copper Cleanup for the Removal of Sulfur from PCB Samples*”, most current revision or replacement.

Attachment I: Air monitoring calculation sheet. Example

Client :						
Date Collected						
Project number						
Project name	NIOSH method 5503 (modified)					
Pace Project #	PCBs in air					
Volume of Air sampled	66.44 liters					
		4034812-007	(Front)	4034812-013	(Back)	
Client Sample ID	Test Parameter	Result in ug	results in ug/L or mg/m3	Result in ug	results in ug/L or mg/m3	<10% breakthrough front to back sections
		Date analyzed: 08/05/10 20:30		Date analyzed: 08/05/10 21:56		
	PCB-1016 (Aroclor 1016)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1221 (Aroclor 1221)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1232 (Aroclor 1232)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1242 (Aroclor 1242)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1248 (Aroclor 1248)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1254 (Aroclor 1254)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1260 (Aroclor 1260)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB-1268 (Aroclor 1268)	<0.044	<0.00066	<0.044	<0.00066	OK
	PCB, Total	<0.044	<0.00066	<0.044	<0.00066	OK
	Tetrachloro-m-xylene (S)	101%	34-130%*	99%	34-130%*	
	Decachlorobiphenyl (S)	96%	34-130%*	95%	34-130%*	
	Samples Extracted:					
	* acceptable range for surrogate recovery					
	Volume of Air sampled (L)	66.44		0.1256	LPM	
	mg/m3 result	0.00066		529	Time	

Result in μg = On column or MDL * final volume of extract ($0.02212 * 2$) = $0.04424 \mu\text{g}$

Attachment II: EPA Method 608, Table 3

Table 3—QC Acceptance Criteria—Method 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for \bar{x} (µg/L)	Range for P, P _s (%)
Aldrin	2.0	0.42	1.08 - 2.24	42 - 122
α-BHC	2.0	0.48	0.98 - 2.44	37 - 134
β-BHC	2.0	0.64	0.78 - 2.60	17 - 147
δ-BHC	2.0	0.72	1.01 - 2.37	19 - 140
γ-BHC	2.0	0.46	0.86 - 2.32	32 - 127
Chlordane	50	10.0	27.6 - 54.3	45 - 119
4,4'-DDD	10	2.8	4.8 - 12.6	31 - 141
4,4'-DDE	2.0	0.55	1.08 - 2.60	30 - 145
4,4'-DDT	10	3.6	4.6 - 13.7	25 - 160
Dieldrin	2.0	0.76	1.15 - 2.49	36 - 146
Endosulfan I	2.0	0.49	1.14 - 2.82	45 - 153
Endosulfan II	10	6.1	2.2 - 17.1	D - 202
Endosulfan Sulfate	10	2.7	3.8 - 13.2	26 - 144
Endrin	10	3.7	5.1 - 12.6	30 - 147
Heptachlor	2.0	0.40	0.86 - 2.00	34 - 111
Heptachlor epoxide	2.0	0.41	1.13 - 2.63	37 - 142
Toxaphene	50.0	12.7	27.8 - 55.6	41 - 126
PCB-1016	50	10.0	30.5 - 51.5	50 - 114
PCB-1221	50	24.4	22.1 - 75.2	15 - 178
PCB-1232	50	17.9	14.0 - 98.5	10 - 215
PCB-1242	50	12.2	24.8 - 69.6	39 - 150
PCB-1248	50	15.9	29.0 - 70.2	38 - 158
PCB-1254	50	13.8	22.2 - 57.9	29 - 131
PCB-1260	50	10.4	18.7 - 54.9	8 - 127

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).

\bar{x} = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).

P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).

D = Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

27. REVISIONS

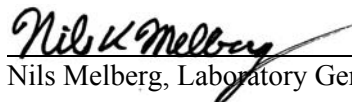
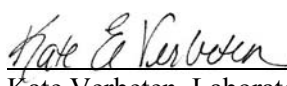

Revision Number	Reason for Change	Date
S-GB-O-047-REV.00	First Issue.	11Sept2008
S-GB-O-047-REV.01	Updated signature page Changed all references to TMX to TCMX Added a note to tables including Aloclor 1262 and 1268 stating they will only be analyzed upon request Section 10.1.1.1: Deleted Aloclor 1262 Section 10.2.3.1: Changed %RSD to %D for surrogate recoveries Section 10.2.5: Added that a low level standard is to be evaluated for each 5 point calibration that is analyzed	09Apr2010
S-GB-O-047-Rev.02	Section 10.1.2.5: Added requirement to verify DDT analogs to not co-elute with Aroclors of interest. Section 12.2 and 12.4: Included language that biota recovery limits should be broadened to 60-130% if laboratory charted limits are narrower than that.	13Sept2010
S-GB-O-047-Rev.03	Section 7.2: Updated analytical and extraction hold times to 365 Days. Section 11.7.3 and Table E: Added Air Wipe Calculations Section 15: Removed SOP reference to Ultrasonic Extraction	14Sept2012
S-GB-O-047-Rev.04	Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 <i>Preparation of SOPs</i> . Updated SOP references throughout document. Throughout Document: Renumbered Tables to match order in document. Added: Tables 6 and 7. Renamed Table E to Attachment I. Sections 11.1.1.1, 12.3.1 and Table 5: Updated AR1660-1 to 0.05 and AR1660-2 to 0.20. Section 11.1.1.3: Updated AR1221 to require a minimum of 3 peaks in lieu of 5.	25Jun2014
S-GB-O-047-Rev.05	Section(s) 11.1.4, 11.1.7.2, 13.4, 13.6.2, 14.1, 25 and Table(s) 6 and Attachment II: Added EPA 608 Method Criteria. Section(s) 11.1.1.3 and 14.1.1: Allow use of 3 peaks for quantitation. Section 12.6.2.1: Removed confirmation by GC/MS. Section 19.5: Added Method Modification for solvent as hexane. Section 26: Added EPA Method 608 Table 3. Throughout Document: Added Uncontrolled Footer Statement.	18Jun2015



STANDARD OPERATING PROCEDURE
DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS
Reference Methods: EPA SW-846 Method 8270C / EPA 625

Local SOP Number:	S-GB-O-049-Rev.07
Effective Date:	Date of Final Signature
Supersedes:	S-GB-O-049-Rev.06
SOP Template Number:	SOT-ALL-O-001-rev.01

APPROVALS

 Nils Melberg, Laboratory General Manager	<u>06/21/17</u> Date
 Kate Verbeten, Laboratory Quality Manager	<u>6/21/17</u> Date
 Chris Haase, Laboratory Department Manager	<u>6/21/17</u> Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Green Bay to determine the concentration of Semi-volatile Organic Compounds (SVOCs) in environmental samples. The laboratory utilizes GC/MS and bases these documented procedures on those listed in EPA SW-846 Method 8270C/ EPA 625. The Green Bay laboratory currently processes water samples by automated separatory funnel using Method SW846 3510C, soil samples by Microwave Extraction using Method SW846 3546 and biota samples by soxhlet extractor using Method SW846 3540C. The latest revision of Pace's SOPs S-GB-O-053 *Separatory Funnel Extraction of Water Samples for Semivolatile Analysis* (most current revision or replacement), S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic hydrocarbon, Base/Neutral/Acids, and Total Petroleum Hydrocarbons on Solid Matrices* (most current revision or replacement), and S-GB-O-033 *Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis* (most current revision or replacement) for these extraction techniques are available from the quality office.

2. Summary of Method

2.1. Sample extracts are prepared for analysis by an appropriate sample preparation method. The semivolatile organic compounds are introduced into the gas chromatograph (GC) by injecting an aliquot of the sample extract. The GC conditions are programmed to separate the analytes. The GC effluent is directly introduced to a mass spectrometer (MS) for both identification and quantification of analytes. Analytes are identified by comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

3. Scope and Application

3.1. This procedure may be used to determine concentrations of neutral, acidic, and basic semivolatile organic compounds in extracts prepared from many types of water samples, soil samples and wastes. Analytes must be soluble in dichloromethane and amenable to capillary gas chromatography. Specific compound classes include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. A list of applicable compounds is shown in Table 11.1 Calibration Standard Compound Concentrations. Pace Reporting Levels (PRLs) are also shown for water and soil samples. PRLs are subject to change based on current analytical system performance and actual sample matrices.

3.2. This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of semi-volatile configured GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.4. This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

4. Applicable Matrices

4.1. This SOP is applicable to soils/sediments, solid wastes, tissue, wipes and aqueous matrices.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is listed in Table 11.1 for the listed methods. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

6.2. Matrix interferences may result from materials co-extracted from some samples.

6.3. Significant phthalate contamination may result at any time if consistent quality control is not practiced. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

6.4. Contamination by carryover can occur when high concentration extracts are analyzed prior to low concentration extracts. The contamination may also cause degradation of labile analytes. Whenever carryover is suspected, the affected extracts should be re-analyzed. If significant degradation of the GC/MS systems is suspected, system performances samples should be analyzed and corrective action taken as needed.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Storage, and Hold time

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One 1L amber glass Samples to be analyzed for EPA 625 must be checked for residual chlorine. If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well.	None	0-<6°C	7 days
Soil/Solid (non-aqueous)	One 8oz wide glass jar	None	0-<6°C	14 days
Biota	-	None	≤ -10°C until extraction	1 year when frozen
TCLP	One 1L Amber Glass	None	0-<6°C	TCLP Leachates must be solvent extracted within 7 days of the completion of the process.
Extracts	2 mL amber glass vials	None	≤ -10°C	40 days

8. Definitions

Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary.

8.1. Toxicity Characteristic Leaching Procedure (TCLP) – An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

8.2. Run Sequence Log – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

8.3. Tune Period – The period after the DFTPP instrument tune check within which analyses may be performed.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 - Instrumentation

Analytical Instrument/Peripherals	EPIC Pro Name	Serial Number
HP 5890 Series II GC	40MSS1	3336A57925
HP 5972 Mass Selective Detector	40MSS1	3501A02320
HP 7673 AutoSampler Tray	40MSS1	3526A39072
HP 7673 Injector	40MSS1	3009A20936
HP Controller	40MSS1	3526A02233
Alcatel 2005 Rough Pump	40MSS1	265402
HP 5890E GC	40MSS6	3310A49571
HP 5972A Mass Selective Detector	40MSS6	3524A03107
HP 18596A AutoSampler Tray	40MSS6	2920A10670
HP 6890 Injector	40MSS6	US0000692
HP 7673 Controller	40MSS6	3113A25880
Edwards E2M2 Rough Pump	40MSS6	53747
Agilent 7890A GC	40MSS8	CN10705029
Agilent 5975C Mass Selective Detector	40MSS8	US71226404
HP Autosampler tray G2614A	40MSS8	US93806114
HP injector G2613A	40MSS8	US93909562
Edwards E2MS Rough Pump	40MSS8	69070
Agilent 7890B	40MSSA	CN15483197
Agilent 5977A Mass Selective Detector	40MSSA	US1422L235
Agilent Autosampler tray G4514A	40MSSA	CN13330090
Agilent injector G4513A	40MSSA	CN14510236
Edwards E2M2 Rough Pump	40MSSA	DUO25

9.2. Table 9.2 - Chromatography Supplies

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	XTI-5 w/ Integraguard	12223-124	30 m, 0.25 mm ID, 0.25 df
Analytical Column	Phenomenex	ZB Semivolatiles Guardian	7HG-G027-11-GGC	20m, 0.18 mm ID, 0.18 df
Fluorocarbon O-rings	Restek		20377	
Vespel/Graphite Ferrules	Restek		20229	1/16" x 0.4 mm ID
Gooseneck Splitless Liner	Restek		20800	4 mm x 6.5 x 78.5 for Aligent GCs
Uniliner	Restek	Drilled Uniler	20771	w/hole in bottom
Inlet Seals	Restek	Dual Vespel Ring Inlet Seals	212389	Stainless steel

9.3. Table 9.3 - Glassware

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	10mL, 25mL, 50mL	Class A
Glass Storage Vials	5mL, 10mL, 12mL, with Teflon-lined screw caps	MG Scientific / T102-3-INV, T102-1-INV V138-19, B510-1
Glass Autosampler Vials	2.0mL with Teflon-lined crimp or screw caps	MG Scientific / V300-3 / V300-20N

9.4. Table 9.4 - General Supplies

Supply	Description	Vendor/Item #
Gas tight syringes	10-μL, 25-μL, 50-μL, 100-μL, 250-μL, 500-μL, and 1,000-μL, as needed, Hamilton or equivalent.	Fisher Scientific/Various
Pipettes	Borosilicate Glass 9" Pipette	MG Scientific / D200-9

10. Reagents and Standards

10.1. Table 10.1 – Reagents

Reagent/Standard	Concentration/ Description	Manufacturer/Vendor/Item #
Methylene Chloride (Dichloromethane)	Pesticide Grade or equivalent / MeCl ₂	MG Scientific / # 9266-8P
Methanol	Purge and Trap Grade or equivalent / MeOH	Burdick & Jackson / VWR Scientific / 232-1
Acetone	Pesticide Grade or equivalent/ Acetone	Burdick and Jackson / 010-4

10.2. Table 10.2 - Standard Definitions

Standard	Description	Comments
Tune Standard	Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine solution in dichloromethane used to verify ion response ratios and system inertness prior to analysis	Must inject no more than 50ng on column
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	1,4 Dichlorobenzene-d4 Naphthalene-d ₈ Acenaphthene-d ₁₀ Phenanthrene-d ₁₀ Chrysene-d ₁₂ Perylene-d ₁₂
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to analysis.	Nitrobenzene-d ₅ 2-Fluorobiphenyl Terphenyl-d ₁₄ Phenol-d ₆ 2-Fluorophenol 2,4,6-Tribromophenol
Spiking Standard	This solution contains 70 target analytes and should not be prepared from the same standards as the calibration standards.	

10.3. Table 10.3 - Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	<ul style="list-style-type: none"> Concentrated reference solution purchased directly from approved vendor 	<ul style="list-style-type: none"> Manufacturer's recommended expiration date for unopened ampulated standards. Stock standards must be replaced 1 year after ampule is opened or on expiration date, whichever is sooner. 	<ul style="list-style-type: none"> Manufacturer's recommended storage conditions When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	<ul style="list-style-type: none"> Reference solutions prepared by dilutions of the stock solution 	<ul style="list-style-type: none"> 1 year from preparation or the expiration date listed for the stock source, whichever is sooner. Working solutions must be checked frequently and replaced if degradation or evaporation is suspected. 	<ul style="list-style-type: none"> Store in amber vials with Teflon lined screw caps <-10 like sample extracts If stock source conditions conflict, store according to method requirements.

10.4. Standard Sources: Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat

materials, recipes for preparing dilutions and working standards, and concentrations for all compounds are presented in table 9.4. All intermediate standards are prepared using dichloromethane and stored in glass vials with Teflon lined caps or as recommended by the standard manufacturer.

10.5. Preparation Procedures:

10.5.1. **Internal Standard Stock solution:** Restek brand Internal Standard Mix, 4000µg/mL, catalog #31006 (contains six internal standards: acenaphthene-d10; chrysene-d12; 1,4-dichlorobenzene-d4; naphthalene-d8; perylene-d12; and phenanthrene-d10), or equivalent. Add 10µL of internal standard solution to 1000µL of every standard, sample, and QC sample injected.

10.5.2. **Surrogate Standard stock solutions:** Restek brand Base Neutral surrogate mix, 5000µg/mL, catalog #31082 (contains four surrogate standards: 2-fluorobiphenyl; nitrobenzene-d5; p-terphenyl-d14; 1,2-Dichlorobenzene-d4 (advisory)). Restek brand Acid surrogate mix, 7500µg/mL, catalog #31083 (contains four surrogate standards: 2-fluorophenol; phenol-d6; 2,4,6-tribromophenol; 2-chlorophenol-d4 (advisory)), or equivalent.

10.5.3. **Surrogate Standard working solution (for extractions):** dilute 5.0mL of both the Restek Base Neutral stock surrogate solution (#31082) and the Restek Acid stock surrogate solution (#31083) to 50mL with Acetone, or equivalent. This gives a final concentration of 500µg/mL per Base Neutral surrogate compound and 750µg/mL per Acid surrogate compound. The extraction analyst spikes each water and soil sample with 100µL of this working solution.

10.5.4. **DFTPP tuning solution:** dilute 1250µL of Supelco DFTPP stock standard (catalog #47548-U; 1000µg/mL) to a total volume of 25mL with methylene chloride for a final concentration of 50µg/mL, or equivalent. The stock standard also contains 4,4'-DDT, benzidine, and pentachlorophenol for assessing column degradation. Information for the standards preparation and expiration dates are affixed to the outside of the vial, and is easily accessible through Epic Pro LIMS. The standard material will be kept in a freezer at -10°C.

10.5.5. **Initial Calibration curve standards:** the following four stock standards, or equivalent, are used to prepare the initial calibration curve:

10.5.5.1 8270 Custom Mix 1, Restek Custom Mix at 200ug/mL cat.#52939

10.5.5.2 1,4-Dioxane, Restek, 2000µg/mL, catalog #30287

10.5.5.3 2,3,4,6-Tetrachlorophenol, AccuStandard, 2000µg/mL, catalog #A-029S-D-10X

10.5.5.4 1,2,4,5-Tetrachlorobenzene, Absolute Standards, 1000µg/mL, catalog #70274

10.5.5.5 **Initial Calibration Intermediate Standard:** Dilute 3mL of 200µg/mL Restek 8270 Custom Mix 1, 300µL of the 2000µg/mL 1,4-Dioxane solution, 300 µL of the 2000 µg/mL 2,3,4,6-Tetrachlorophenol solution, and 600 µL of 1000µg/mL 1,2,4,5-Tetrachlorobenzene to 5.0mL with dichloromethane, or equivalent. The resulting intermediate standard has a concentration of 120mg/L for each compound.

10.5.6. **Working Standard Preparation:** Working calibration standards are prepared in dichloromethane or a water soluble solvent. Standards made for direct analysis on the GC/MS are made in dichloromethane. Standards made for addition into samples as part of the preparation are made into Acetone. Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks or prepared in smaller, glass vials and brought to volume by additions of solvent with micro syringes.

10.5.6.1 **Initial Calibration Verification stock standards (second-source)**

10.5.6.1.1 O2si, 200ug/mL, Catalog #113881-05.

10.5.6.1.2 n-Nitrosodiphenylamine, Supelco, 5000ug/mL, Catalog #46702-U.

10.5.6.1.3 Supelco, 1,4-Dioxane, catalog #48367, 2000µg/mL.

10.5.6.1.4 Absolute Standards, 2,3,4,6-tetrachlorophenol, catalog #92389, 5000µg/mL, or equivalent.

10.5.6.1.5 Supelco, 1,2,4,5-Tetrachlorobenzene, catalog #40177, 1000µg/mL.

10.5.6.2 **Initial Calibration Verification working standard (second source):**

Dilute 250µL of the custom 8270 second source standard #113881-05, 10µL of 2,3,4,6-Tetrachlorophenol #560028, 25µL of 1,4-Dioxane #48367, 50µL of 1,2,4,5-Tetrachlorobenzene #40177, 10uL of 5000ug/mL n-Nitrosodiphenylamine, 10uL of 5000ug/mL B/N surrogate mix, 6.7uL of 7500ug/mL Acid surrogate mix and 10µL of the stock internal standard solution (9.5.1) to 1mL with dichloromethane, or equivalent. This gives a final concentration of 50ppm.

10.5.6.3 **LCS/MS Standard working solution:** Supelco 70 Component Custom MCS Mix catalog #861389-U, 200µg/mL. Supelco n-Nitrosodiphenylamine, catalog #46702-U, 5000µg/mL, or equivalent. The extraction analyst spikes each LCS/LCSD and matrix spike sample with 250µL of the LCS mix and 10µL of the n-NDPA solution. This produces a concentration of 50µg/mL.

10.5.6.4 **Other calibrations:** Other compounds are analyzed per client requests. Curves are prepared at levels similar to those of the standards above. The calibration standards and the second source standards are as follows: Calibration Standards, Benzidine, Calibration Standard, Supelco Catalog #40005, 5000µg/mL. Second Source, Restek, catalog #31441, 1000µg/mL; EPA CLP SOW OLM4 mix, Calibration Standard, Supelco Catalog#47514-U, 2000µg/mL. Second Source, Absolute Standards, catalog #19253, 2000µg/mL, or equivalent. Minnesota Phenols Samples required Calibration Standard Supelco 500ug/mL, cat.#LC12745, o2si, catalog #114055-05, 500 µg/mL. Phenol and 345-trichlorophenol, first source, Absolute Standards, second source; o2Si

10.5.6.5

10.5.7. Store at -10°C or less in amber Teflon-sealed containers. The solutions should be checked frequently for stability.

10.6. **Calibration Standard Preparation:** Calibration standards are made into dichloromethane for the purpose of direct analysis by the analytical instrumentation. The standards must be made in a volumetric fashion. Several alternatives exist but the method employed by Pace – Green Bay utilizes glass autosampler vials according to the following procedure. The individual standards can be made according to the details provided in table 10.3.

Table 10.4 – Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Calibration Std 1	41.5µL	Dichloromethane	958.5µL	1010µL	5ppm
Calibration Std 2	83µL	Dichloromethane	917µL	1010µL	10ppm
Calibration Std 3	209µL	Dichloromethane	791µL	1010µL	25ppm
Calibration Std 4	417µL	Dichloromethane	583µL	1010µL	50ppm
Calibration Std 5	667µL	Dichloromethane	333µL	1010µL	80ppm
Calibration Std 6	833µL	Dichloromethane	167µL	1010µL	100ppm
Calibration Std 7	1000µL	Dichloromethane	0µL	1010µL	120ppm
Continuing Calibration Verification Standard	417µL	Dichloromethane	583µL	1010µL	50ppm

10.7. Traceability of Calibration Standards—The calibration standards purchased from vendors have been manufactured according to the following guidelines

10.7.1. Identity of neat material verified by GC/MS

10.7.2. Purity of neat material determined by GC/FID or GC/ECD. Correction for impurities is made when purity is less than 97%. Standards are prepared gravimetrically to a precision of 0.5%. All weights are traceable to NIST.

10.7.3. Analyte concentration verified by capillary gas chromatography. Standards tested for stability and homogeneity.

10.7.4. Standards are expiration dated.

10.8. Standard Labeling—All working calibration standards will have a label attached to the bottle identifying the following (Epic pro standard labels do not contain all the following)

10.8.1. Name of Solution

10.8.2. PASI, LLC. Standard ID Number

10.8.3. PASI, LLC. Lab Lot ID (for Stock standards and reagents)

10.8.4. Preparation Date

10.8.5. Preparer's initials

10.8.6. Concentration

10.8.7. Expiration Date

11. Calibration and Standardization

11.1. Tune Verification – The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method (see section 12.2), follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures. Refer to section 12.2 for details on the analysis and evaluation of this standard.

11.2. Initial Calibration:

11.2.1. Analysis of Standards: An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 11.1 for compound concentrations.

Table 11.1: Laboratory PQL and Calibration Standard Compound Concentrations

Analyte	PQL water (µg/L)	PQL soil (µg/kg)	PQL Biota (µg/kg)	Std 1 µg/L	Std 2 µg/L	Std 3 µg/L	Std 4 µg/L	Std 5 µg/L	Std 6 µg/L	Std 7 µg/L
Acenaphthene	5.0	167	330	5.0	10	25	50	80	100	120
Acenaphthylene	5.0	167	330	5.0	10	25	50	80	100	120
Aniline	5.0	167	N/A	5.0	10	25	50	80	100	120
Anthracene	5.0	167	330	5.0	10	25	50	80	100	120
Benz(a)anthracene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(a)pyrene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(b)fluoranthene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(g,h,i)perylene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(k)fluoranthene	5.0	167	330	5.0	10	25	50	80	100	120
Benzoic acid	10	330	N/A	5.0	10	25	50	80	100	120
Benzyl alcohol	10	330	N/A	5.0	10	25	50	80	100	120
4-Bromophenylphenyl ether	5.0	167	330	5.0	10	25	50	80	100	120
Butylbenzylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
Carbazole	5.0	167	330	5.0	10	25	50	80	100	120
4-Chloro-3-methylphenol	5.0	167	330	5.0	10	25	50	80	100	120
4-Chloroaniline	10	333	330	5.0	10	25	50	80	100	120
bis(2-Chloroethoxy)methane	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Chloroethyl) ether	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Chloroisopropyl) ether	5.0	167	330	5.0	10	25	50	80	100	120
2-Chloronaphthalene	5.0	167	330	5.0	10	25	50	80	100	120
2-Chlorophenol	5.0	167	330	5.0	10	25	50	80	100	120
4-Chlorophenylphenyl ether	5.0	167	330	5.0	10	25	50	80	100	120
1,2-Diphenylhydrazine	5.0	167	N/A	5.0	10	25	50	80	100	120
Chrysene	5.0	167	330	5.0	10	25	50	80	100	120
Dibenz(a,h)anthracene	5.0	167	330	5.0	10	25	50	80	100	120
Dibenzofuran	5.0	167	330	5.0	10	25	50	80	100	120
1,2-Dichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
1,3-Dichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
1,4-Dichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
3,3'-Dichlorobenzidine	10	330	330	5.0	10	25	50	80	100	120
2,4-Dichlorophenol	5.0	167	330	5.0	10	25	50	80	100	120
Diethylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
2,4-Dimethylphenol	5.0	167	330	5.0	10	25	50	80	100	120
Dimethylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
Di-n-butylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
4,6-Dinitro-2-methylphenol	5.0	333	670	5.0	10	25	50	80	100	120
2,4-Dinitrophenol	10	333	670	5.0	10	25	50	80	100	120
2,4-Dinitrotoluene	5.0	167	330	5.0	10	25	50	80	100	120
2,6-Dinitrotoluene	5.0	167	330	5.0	10	25	50	80	100	120
Di-n-octylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Ethylhexyl)phthalate	5.0	167	330	5.0	10	25	50	80	100	120
Fluoranthene	5.0	167	330	5.0	10	25	50	80	100	120
Fluorene	5.0	167	330	5.0	10	25	50	80	100	120
Hexachloro-1,3-butadiene	10	333	330	5.0	10	25	50	80	100	120
Hexachlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
Hexachlorocyclopentadiene	5.0	167	330	5.0	10	25	50	80	100	120
Hexachloroethane	5.0	167	330	5.0	10	25	50	80	100	120
Indeno(1,2,3-cd)pyrene	5.0	167	330	5.0	10	25	50	80	100	120
Isophorone	5.0	167	330	5.0	10	25	50	80	100	120

Analyte	PQL water (µg/L)	PQL soil (µg/kg)	PQL Biota (µg/kg)	Std 1 µg/L	Std 2 µg/L	Std 3 µg/L	Std 4 µg/L	Std 5 µg/L	Std 6 µg/L	Std 7 µg/L
2-Methylnaphthalene	5.0	167	330	5.0	10	25	50	80	100	120
2-Methylphenol	10	333	330	5.0	10	25	50	80	100	120
3&4-Methylphenol	5.0	167	330	5.0	10	25	50	80	100	120
Naphthalene	5.0	167	330	5.0	10	25	50	80	100	120
2-Nitroaniline	5.0	167	330	5.0	10	25	50	80	100	120
3-Nitroaniline	5.0	167	670	5.0	10	25	50	80	100	120
4-Nitroaniline	10	333	670	5.0	10	25	50	80	100	120
Nitrobenzene	5.0	167	330	5.0	10	25	50	80	100	120
2-Nitrophenol	5.0	167	330	5.0	10	25	50	80	100	120
4-Nitrophenol	10	333	670	5.0	10	25	50	80	100	120
N-Nitrosodimethylamine	5.0	167	330	5.0	10	25	50	80	100	120
N-Nitroso-di-n-propylamine	5.0	167	330	5.0	10	25	50	80	100	120
N-Nitrosodiphenylamine	5.0	333	330	5.0	10	25	50	80	100	120
Pentachlorophenol	10	330	670	5.0	10	25	50	80	100	120
Phenanthrene	5.0	167	330	5.0	10	25	50	80	100	120
Phenol	5.0	167	330	5.0	10	25	50	80	100	120
Pyrene	5.0	167	330	5.0	10	25	50	80	100	120
Pyridine	5.0	167	330	5.0	10	25	50	80	100	120
1,2,4-Trichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
2,4,5-Trichlorophenol	5.0	167	670	5.0	10	25	50	80	100	120
2,4,6-Trichlorophenol	5.0	167	330	5.0	10	25	50	80	100	120
1,4-Dioxane	10	330	N/A	5.0	10	25	50	80	100	120
1,2,4,5-Tetrachlorobenzene	5.0	167	N/A	5.0	10	25	50	80	100	120
2,3,4,6-Tetrachlorophenol	10	167	N/A	5.0	10	25	50	80	100	120
Acetophenone	10	333	N/A	5.0	10	25	50	80	100	N/A
Atrazine	10	333	N/A	5.0	10	25	50	80	100	N/A
Benzaldehyde	10	333	N/A	5.0	10	25	50	80	100	N/A
Benzidine	50	1670	N/A	5.0	10	25	50	80	100	N/A
Caprolactam	10	333	N/A	5.0	10	25	50	80	100	N/A
Biphenyl	10	333	N/A	5.0	10	25	50	80	100	N/A

11.2.2. An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 12.15. Results for these TICs should be reported only on a present/absent basis. However, quantitative results may be reported provided they are qualified as estimated values.

11.2.3. Calibration Response Factors: Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

11.2.4. To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound

relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard (µg/L).

C_x = Concentration of the compound being measured (µg/L).

11.2.5. Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8270C method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples.

11.2.6. **Calibration Curve Fit:** The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the “goodness of fit”.

11.2.7. **Average Response Factor (ARF):** The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF (see Table 11.2):

$$\%RSD = \left(\frac{SD \times 100}{ARF} \right)$$

Where:

SD = Standard deviation of the averaged RFs for a given compound

11.2.8. The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The **Calibration Check Compounds (CCCs)** are a subset of the target analyte list that must meet specific criteria (see Table 11.2) for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria. If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated.

11.2.9. **Linear Regression:** The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y=ax+b$ where “a” is the slope of the line and “b” is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y axis at

the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient “r” is used to determine the acceptability of a linear regressed curve (see Table 11.2).

11.2.10. Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y = ax^2 + bx + c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

11.2.11. A calculation of the coefficient of determination (COD) is used to determine the acceptability of a non-linear regressed curve (see Table 11.2). Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented and permission of the supervisor or the quality manager is obtained. The point must be dropped in its entirety. Re-analysis if performed should be within the same 12 hour time window and must occur within 8 hours of the original analysis.

11.3. Calibration Verification:

11.3.1. Low Level Calibration Check (CRDL): The lowest range of the calibration will be checked by either refitting the lowest calibration point against the calibration curve or re-analyzing the lowest calibration point. The CRDL must be checked before running any sample from MN and must meet a recovery of 60-140% of the expected value. Any compounds failing must be flagged in MN samples as failing to meet CRDL limits.

11.3.2. Second Source Verification: In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the “fit” of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

11.3.3. Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification (ICV)**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the ICV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\% \text{ Difference} = \frac{(RF_{CCV} - AveRF_{cal})}{AveRF_{cal}} * 100$$

$$\% \text{ Drift} = \frac{(\text{Result CCV} - \text{True Value CCV})}{\text{True Value CCV}} * 100$$

11.3.4. **Continuing Calibration Verification (CCV)**: As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished by analyzing a midpoint calibration standard (CCV).

11.3.5. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

11.3.6. The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\% \text{ Difference} = \frac{(RF_{CCV} - AveRF_{cal})}{AveRF_{cal}} * 100$$

$$\% \text{ Drift} = \frac{(\text{Result CCV} - \text{True Value CCV})}{\text{True Value CCV}} * 100$$

Table 11.2: Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD \leq 15%	If not met, try linear regression fit
	Linear Regression	$r \geq 0.99$	If not met, try non-linear regression fit
	Non-linear Regression	COD ≥ 0.99	If not met, remake standards and recalibrate
System Performance Check Compounds (SPCCs)	N-Nitroso-di-n-propylamine Hexachlorocyclopentadiene 2,4-Dinitrophenol 4-Nitrophenol	Avg RF ≥ 0.05 Avg RF ≥ 0.05 Avg RF ≥ 0.05 Avg RF ≥ 0.05	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system.
Calibration Check Compounds (CCCs)	Acenaphthene 1,4-Dichlorobenzene Hexachlorobutadiene N-Nitrosodiphenylamine Di-n-octylphthalate Fluoranthene Benzo[a]pyrene 4-Chloro-3-methylphenol 2,4-Dichlorophenol 2-Nitrophenol Phenol Pentachlorophenol 2,4,6-Trichlorophenol	%RSD $< 30\%$	%RSD for the calibration check compounds (CCC's) must be $\leq 30\%$ regardless of curve fit used. If the CCCs are not included on a list of analytes for a project, and therefore not included in the calibration standards, then all compounds of interest must meet a $\leq 15\%$ RSD criterion.
Second Source Verification Standard	Immediately after each initial calibration	% Drift $\pm 30\%$	Acceptance criteria are $\pm 30\%$ for all analytes, with allowances for 5% of compounds at $\pm 40\%$. See current revision of S-GB-Q-026. Additional client specific requirements for the analysis of contract samples requires that all compounds must be within $\pm 20\%$.
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT	RT ± 30 sec	Use midpoint calibration standard as reference
	Internal Standard Response	50 – 200%	Use midpoint calibration standard as reference
	CCCs	RF $\pm 20\%$ Diff. Result $\pm 20\%$ Drift	Use for Avg RF calibration curves Use for linear and non-linear calibration curves
	Non-CCC Targets	EPA 8270 Criteria: RF $\pm 50\%$ Diff. Result $\pm 50\%$ Drift EPA 625 Criteria: RF $\pm 20\%$ Diff. Result $\pm 20\%$ Drift	Some programs may require control over non-CCC target analytes. In the absence of specified criteria, use those listed

11.4. Calibration Corrective Actions:

11.4.1. Calibration Linearity Problems:

- 11.4.1.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.
- 11.4.1.2 Perform another initial calibration.
- 11.4.1.3 No data can be reported.
- 11.4.1.4 Generate Non-Conformance Memo.

11.4.2. Second Source Verification Problems:

- 11.4.2.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.
- 11.4.2.2 Perform another initial calibration.
- 11.4.2.3 No data can be reported.
- 11.4.2.4 Generate Non-Conformance Memo.

11.4.3. Continuing Calibration Verification Problems:

- 10.4.3.1. Reanalyze the original CCV standard to determine instrument consistency.
- 10.4.3.2. Prepare and analyze a new CCV standard to determine preparation consistency/standard integrity.
- 10.4.3.3. Document instrument maintenance.
- 10.4.3.4. Reanalyze CCV standard to determine if maintenance was effective in restoring performance.
- 10.4.3.5. Complete recalibration of instrument.
- 10.4.3.6. If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the injection log with a thorough explanation for the deviation from policy.
- 10.4.3.7. Exceptions: If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

12. Procedure

12.1. Operating Parameters: Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 12.1: Instruments and Operating Parameters

GC/MS Instrument 40MSS1	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 40°C	Acquisition mode: SCAN
Temp Program: hold 1.0 min at 40°C, ramp at 18°C/min to 100°C, then ramp at 15°C/min to 290°C, hold 5.95min, then ramp at 40°C/min to 320°C and hold for 1 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Restek XTI-5 (30m; 0.25mm ID and 0.25µm film thickness)w/Integruguard	
Purge Flow: 40mL/min	
GC/MS Instrument 40MSSA	
GC: Agilent 7890B	MS: Hewlett Packard model 5977A
Operating Parameters:	Operating Parameters:
Initial Temp: 45°C	Acquisition mode: SCAN
Temp Program 45°C hold 1.00min, ramp at 30°C/min to 260°C hold for 0min, then ramp at 6°C/min to 295°C and hold for 0 min, then ramp at 25C/min to 325C and hold for 2min	Mass Range: 35-550
Final Temp: 325°C	
Transfer Line Temp: 300°C	
Column: Phenomenex ZB-Semivolatile Guardian 30 m, 0.25 ID(mm), 0.25 film thickness(mm)	
Split Ratio: L/minb 10:1	
GC/MS Instrument 40MSS8	
GC: Agilent 7890A	MS: Hewlett Packard model 5975
Operating Parameters:	Operating Parameters:
Initial Temp: 45°C	Acquisition mode: SCAN
Temp Program 45°C hold 1.00min, ramp at 30°C/min to 260°C hold for 0min, then ramp at 6°C/min to 295 °C and hold for 0 min,then ramp 25C/min to 325C and hold for 2 min.	Mass Range: 35-550
Final Temp: 325°C	
Transfer Line Temp: 300°C	
Column: Phenomenex ZB-Semivolatile Guardian 30 m, 0.25 ID(mm), 0.25 film thickness(mm)	
Split Ratio: 10:1	
GC/MS Instrument 40MSS6 used for Minnesota Phenols Samples	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 50°C	Acquisition mode: SCAN
Temp Program 50°C, ramp at 18°C/min to 150°C, then ramp at 3°C/min to 167°C , then ramp at 40°C/min to 320°C and hold for 2.5 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Phenomenex ZB-Semivolatiles (30m; 0.25µm ID, 0.25 df)	
Split Flow: 100mL/min	

12.2. Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing DFTPP. The tune verification standard can be combined with the CCV standard provided that the amount of DFTPP introduced into the system meets the method criteria. For semi-volatile analysis, the system must also be verified for inertness. This is done simultaneously by the inclusion of DDT, benzidine and pentachlorophenol. DDT is used to verify breakdown conditions; benzidine and pentachlorophenol are used to check for tailing due to system activity.

12.2.1. After the analysis of this standard, the mass spectrum of DFTPP must be evaluated against the following criteria:

Mass (m/z)	Ion Abundance criteria
51	10.0-80.0% of m/z 198
68	<2.0% of m/z 69
69	Present
70	<2.0% of m/z 69
127	10.0-80.0% of m/z 198
197	<2.0% of m/z 198
198	Base peak, >50% of Mass 442
199	5.0-9.0% of m/z 198
275	10.0-60.0% of m/z 198
365	>1% of m/z 198
441	Present, but less than m/z 443
442	>50.0% of m/z 198
443	15.0-24.0% of m/z 442

12.2.2. To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for DFTPP. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one of the following manners, in the listed order:

- 1. Using an average of three scans, centered on the apex of the peak; or,**
- 2. Using an average of all scans across the width of the peak, taken at half height; or,**
- 3. Using an average of all scans taken across the width of the peak from baseline to baseline.**

A background scan taken immediately before but not including the peak must be subtracted.

12.2.3. Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the DFTPP tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the DFTPP tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.?

12.2.4. If the ratios do not meet the criteria, refer to the following corrective actions to address the problem: Any changes made to the system must be followed with the reanalysis of a tune verification standard. Any maintenance performed on the physical mass spec components requires recalibration. "Autotunes" may be performed as long as the following CCV meets all criteria for response, retention time and sensitivity.

12.3. Tailing Factor Verification- Benzidine and Pentachlorophenol should be present at their normal responses, and peak tailing should not be to an excess.

12.3.1. **Column performance test for base / neutrals** – At the beginning of each day that the base / neutral fraction is to be analyzed for benzidine, the benzidine tailing factor must be calculated. The benzidine tailing factor must be less than 3.0.

12.3.2. **Column performance test for acids** – At the beginning of each day that the acid fraction is to be determined, the pentachlorophenol tailing factor must be calculated. The pentachlorophenol tailing factor must be less than 5.0.

12.3.3. **Tailing factor calculation** – Refer to Attachment II: Tailing Factor Calculation.

12.3.4. The tailing factor of 3.0 for Benzidine and 5.0 for Pentachlorophenol must not be exceeded. If the tailing factor for either exceeds this amount, corrective action must be taken prior to the analysis of samples(unless all compounds required by samples analyzed after this tune and check meet Calibration Check Compound(CCC) limits). The tailing factor must be verified by the analysis of another tailing factor standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

12.3.4.1 Perform front-end maintenance on the GCMS System.

12.3.4.2 Begin the run again by re-analyzing the DFTPP tune solution.

12.4. Breakdown Verification- The GC/MS system must be sufficiently inert such that DDT will not breakdown excessively while in the injection port. The inertness is assessed by calculating the percent breakdown of DDT into the products DDD and DDE. The calculation is performed as follows:

$$\% \text{DDT Breakdown} = \left(\frac{(\text{DDD} + \text{DDE})}{(\text{DDT} + \text{DDD} + \text{DDE})} \right) * 100$$

12.4.1. The % breakdown **must not exceed 20%**. If the breakdown of DDT exceeds this amount, corrective action must be taken prior to analysis of samples(unless all compounds required by samples analyzed after this tune and check meet Calibration Check Compound(CCC) limits). The breakdown must be verified by the analysis of another breakdown standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

12.4.1.1 Perform front-end maintenance on the GCMS System

12.4.1.2 Begin the run again by re-analyzing the DFTPP tune solution.

12.5. Calibration Verification: After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 11.2 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

12.6. If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 11.4.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

12.7. Note: In situations where the instrument will run unattended (i.e., overnight), the analyst may load sequential CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, the CCV must be evaluated according to the protocol set forth in the Quality Assurance Manual within the Equipment and Measurement Traceability section.

12.8. Sample Preparation-

12.8.1. **Water Samples:** Aqueous samples are prepared according to EPA 3510C. These procedures are contained in a separate standard operating procedure. Refer to SOP number S-GB-O-053 *Separatory Funnel Extraction of Water Samples for Semivolatile* (most current revision or replacement) for details on the preparation of aqueous samples.

12.8.1.1 Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10µL of the internal standard solution.

12.8.2. **Soil Samples:** Solid samples are prepared according to EPA 3546. These procedures are contained in a separate standard operating procedure. Refer to SOP number S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices* (most current revision or replacement) for details on the preparation of soil or solid samples.

12.8.2.1 Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10µL of the internal standard solution.

12.8.3. **Biota Samples:** Biota samples are prepared according to EPA Method 3540C. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-033 *Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis* (most current revision or replacement) for details on the preparation of biota samples.

12.8.3.1 Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 5µL of the internal standard solution.

12.9. Dilutions

12.9.1. Dilutions on sample extracts must be prepared in a volumetric fashion. Sample aliquots should be taken in volumetric syringes and brought to volume by the addition of solvent via an appropriate syringe. In the event a dilution is made to bring a target analyte into calibration range, the analyst should make a dilution such that the target analyte is roughly the equivalent of the mid calibration point whenever possible. If dilutions are made on extracts that already contain internal standards, a proportional aliquot of internal standard solution must be added to the diluted extract based on the volume of diluent used.

12.10. Sample Analysis-

12.10.1. GC/MS System Preparation

12.10.1.1 Operating Parameters – Set up the instrument parameters shown in Table 12.1

12.10.1.2 System Tuning and GC Performance Checks – Analyze the Tuning Solution and tune the mass spectrometer to meet the criteria shown in Section 12.2. Verify acceptable GC system performance as described in Section 12.2. Print out a tune report.

12.10.1.3 Batch Sequence – Generate a sequence to run a batch of samples.

Initial Calibration – The typical batch for initial calibration should include:

Tune Standard
Calibration Level 1
Calibration Level 2
Calibration Level 3
Calibration Level 4
Calibration Level 5
Calibration and System Performance Solution

Sample Analysis – The typical batch for sample analysis should include the following. Preparation of LCS, MS, MSD, and Duplicate sample extracts is described in the appropriate sample preparation SOP.

Tune Standard
Calibration and System Performance Solution
Instrument Blank
Method Blank
Laboratory Control Sample
Laboratory Control Sample Duplicate
20 samples
Matrix Spike
Matrix Spike Duplicate

Autosampler – Load the Autosampler with standards and samples for the batch created above.

12.10.1.4 Analyze Samples – Analyze all standards, quality control samples, and environmental samples.

12.10.1.5 Process all runs with Target software

12.10.1.6 View sample chromatograms and verify analyte identifications (Section 12.11).

12.10.1.7 Post data to EPIC Pro.

12.11. Data Reduction

12.11.1. Qualitative Analysis: This must be done on every sample and quality control standard.

12.11.1.1 **Retention Time Comparison:** The relative retention time (RRT) of the sample component must be within ± 0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co eluting components.

12.11.1.2 **Mass Spectrum Comparison:** The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.
- Additional client specific requirements for the analysis of contract samples requires all ions present in the reference mass spectrum at a relative intensity > 10% must be present in the sample spectrum.
- Due to limitations of the “Target” software, analyst discretion is advised.

Table 12.2 Primary and Secondary quantitation ions for target compounds²

Analyte	Primary Ion	Secondary Ions
Phenol	94	65, 66
Bis (2-Chloroethyl) ether	63	93, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl Alcohol	108	79, 107
1,2-Dichlorobenzene	146	148, 111
2-Methylphenol	108	107, 79
Bis (2-Chloroisopropyl)ether	45	77, 121
3&4-Methylphenol	108	107, 79
N-Nitroso-di-n-propylamine	70	43, 101
Hexachloroethane	117	201, 199
1,4-Dioxane	88	58, 43
Benzaldehyde	105	106, 77
N-Nitrosodimethylamine	42	74
Aniline	93	66, 39
Acetophenone	105	77, 120
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	107	122, 121
Benzoic acid	122	105, 77
Bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Caprolactam	55	56, 113
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	153
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	108, 92
Acenaphthene	154	153, 152
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65, 39
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 89
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	108, 92
1,2,4,5-Tetrachlorobenzene	216	214, 179
2,3,4,6-Tetrachlorophenol	232	131, 166
4,6-Dinitro-2-methylphenol	198	51, 105

Analyte	Primary Ion	Secondary Ions
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	283.7	142, 249
Atrazine	200	173, 58
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	185, 92
Pyrene	202	200, 203
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
Chrysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
Di-n-octylphthalate	149	167, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 277
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Carbazole	167	168, 169

²The information in this table was taken from Method 8270C. Please refer to the method for additional compounds and their applicable ions.

Analyte	Primary Ion	Secondary Ions
Internal Standards		
1,4-Dichlorobenzene-d ₄	152	150, 115
Naphthalene-d ₈	136	68
Acenaphthene-d ₁₀	164	162, 160
Phenanthrene-d ₁₀	188	94, 80
Chrysene-d ₁₂	240	120, 236
Perylene-d ₁₂	264	260, 265
Surrogates		
2-Fluorophenol (acid)	112	64
Phenol-d ₆ (acid)	99	71
Nitrobenzene-d ₅ (BN)	82	128, 54
2-Fluorobiphenyl (BN)	172	171
2,4,6-Tribromophenol (acid)	329.8	331.8, 141
Terphenyl-d ₁₄ (BN)	244	122, 212

²The information in this table was taken from Method 8270C. Please refer to the method for additional compounds and their applicable ions.

12.11.2. Internal Standard Assignment List (from Method SW-846 8270C-Table 5): this section lists the internal standard compounds and all target compounds that are assigned to each internal for quantitation:

1,4-Dichlorobenzene – d4

Aniline
Benzyl alcohol
Bis (2-chloroethyl)ether
Bis(2-chloroisopropyl)ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
2-Fluorophenol (surrogate)
Hexachloroethane
2-Methylphenol
4-Methylphenol
N-Nitroso-dimethylamine
N-Nitroso-di-n-propylamine
Phenol
Phenol-d6 (surrogate)
1,4-Dioxane
Pyridine
2-Chlorophenol-d4 (advisory surrogate)
Benzaldehyde
1,2-Dichlorobenzene-d4 (advisory surrogate)

Acenaphthene-d10

Acenaphthene
Acenaphthylene
1,2-Diphenylhydrazine
2-Chloronaphthalene
4-Chlorophenyl phenyl ether
Dibenzofuran
Diethyl phthalate
Dimethyl phthalate
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Fluorene
2-Fluorobiphenyl (surrogate)
Hexachlorocyclopentadiene
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
4-Nitrophenol
Biphenyl
2,4,6-Tribromophenol (surrogate)
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
1,2,4,5-Tetrachlorobenzene

Naphthalene-d8

Acetophenone
Benzoic acid
Bis(2-chlorethoxy)methane
4-Chloroaniline
4-Chloro-3-methylphenol
2,4-Dichlorophenol
2,6-Dichlorophenol
2,4-Dimethylphenol
Hexachlorobutadiene
Isophorone
2-Methylnaphthalene
Naphthalene
Nitrobenzene
Nitrobenzene-d8 (surrogate)
2-Nitrophenol
1-Methylnaphthalene
1,2,4-Trichlorobenzene

Phenanthrene-d10

Atrazine
Anthracene
4-Bromophenyl phenyl ether
Di-n-butyl phthalate
4,6-Dinitro-2-methylphenol
Carbazole
Fluoranthene
Hexachorobenzene
N-Nitroso-diphenylamine
Pentachlorophenol
Phenanthrene

Chrysene-d12

Benzidine
Benzo(a)anthracene
Bis(2-ethylhexyl)phthalate
Butyl benzyl phthalate
Chrysene
3,3'-Dichlorobenzidine
Pyrene
Terphenyl-d6 (surrogate)
Di-n-octylphthalate

Perylene-d12

Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Dibenz(a,h)anthracene
Indeno(123,cd)pyrene

12.12. Quantitative Analysis- Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

12.12.1. **Raw Data Results:** The GC/MS data system will calculate the concentration of each analyte as µg/L (or ng/mL). For water samples, no further calculations are necessary unless a dilution of the sample has been performed. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

12.13. **Tentatively Identified Compounds (TICs)** – For some samples, identification may be desired for non-target compounds. A mass spectral library search may be conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

12.13.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum;

12.13.2. The relative intensities of the major ions should agree within $\pm 20\%$;

12.13.3. Molecular ions present in the reference spectrum should be present in the sample spectrum;

12.13.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds;

12.13.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.13.6. For additional information on the determination of TICs, please see SOP: S-ALL-O-038, *Processing of TICs for GCMS* (most current revision or replacement).

13. Quality Control

13.1. Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Target analytes must be less than reporting limit.	Qualify results and/or re-extract associated samples. <u>Exceptions:</u> If sample ND, report sample without qualification; If sample result >10x MB detects, report sample with appropriate qualifier indicating blank contamination; If sample result <10x MB detects, and sample cannot be re-extracted, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Method specified compounds: Base Neutrals: 1,2,4-Trichlorobenzene; Acenaphthene; 2,4-Dinitrotoluene; Pyrene; N-nitroso-di-n-propylamine; 1,4-Dichlorobenzene Acids: Pentachlorophenol; Phenol; 2-Chlorophenol; 4-Chloro-3-methylphenol; 4-Nitrophenol <u>OR (alternative)</u> 70 compound LCS Mix	One per batch of up to 20 samples	Laboratory derived limits <u>Method Specified List:</u> All compounds must pass control criteria, with no exceptions. <u>Full Target List:</u> Marginal exceedances allowed according to the TNI standard.	At analyst discretion, Re-analyze the LCS to verify failure; If LCS passes, review samples for potential injection problems; If problem persists, check spike solution; Re-extract samples where possible. <u>Exceptions:</u> If LCS recovery is > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. Also, if the MS/MSD meet QC requirements, they may be used as acceptable criteria for the LCS.
Matrix Spike (MS)	Method specified compounds: Base Neutrals: 1,2,4-Trichlorobenzene; Acenaphthene; 2,4-Dinitrotoluene; Pyrene; N-nitroso-di-n-propylamine; 1,4-Dichlorobenzene Acids: Pentachlorophenol; Phenol; 2-Chlorophenol; 4-Chloro-3-methylphenol; 4-Nitrophenol <u>OR (alternative)</u> 70 Compounds LCS Mix	One per batch of up to 20 samples EPA 625: One per batch of up to 10 samples	Laboratory derived limits <u>Method Specified List:</u> All compounds must pass control criteria, with no exceptions. <u>Full Target List:</u> Marginal exceedances allowed according to the TNI standard.	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate <u>OR (alternative)</u> Sample Dup	One for every 5% of all environmental samples EPA 625: One for every 10% of all environmental samples	Laboratory Derived Limits	Report results with an appropriate footnote.

13.2. Table 13.2 – Sample Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	Retention Time: RT must be ± 30 seconds from last calibration check on all samples	Retention Time Failure: If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting; Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	Nitrobenzene-d5 2-Fluorobiphenyl Terphenyl-d14 Phenol-d6 2-Fluorophenol 2,4,6-Tribromophenol	Added to all samples, spikes, control samples and method blanks prior to analysis	Laboratory derived limits	1 Base neutral and 1 Acid surrogate are allowed to be outside of recovery limits before action is taken. Assess impact of sample matrix. In the absence of obvious matrix interference (high background, extremely dark extract), re-extract sample. <u>Exceptions:</u> Surrogate recovery above criteria and target compounds < RL, result may be reported with appropriate footnote. Surrogate recovery out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote.

14. Data Analysis and Calculations

14.1. Results Calculation- Aqueous Samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(C_x)(V_x)(DF)}{(V_s)}$$

Where:

C_x = Concentration in extract (μg/mL).
 V_v = Volume of final extract (mL).
 DF = Dilution factor.
 V_s = Volume of water sample extracted (mL).

14.2. Results Calculation- Soil/Solid Samples:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(C_x)(V_x)(1000)(DF)}{(W_s)}$$

Where:

C_x = Concentration in extract (μg/mL).
 V_v = Volume of final extract (mL).
 DF = Dilution factor.
 W_s = Weight of soil sample extracted (g).

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Tables 11.2, 13.1, and 13.2.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Tables 11.2, 13.1, and 13.2

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Tables 11.2, 13.1, and 13.2.

18. Method Performance

18.1. **Method Detection Limit (MDL) Study:** An MDL study must be conducted annually per S-GB-Q-020, *Determination of LOD and LOQ* (most current revision or replacement) for each matrix per instrument.

18.2. **Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures*, (most current revision or replacement).

18.2.1. Analysis of four (4) replicates of reagent water spiked with 250µL of the 8270 LCS Spiking Solution and 10µL of nNPDA plus all other compounds that are currently reported at a concentration of 50µg/L or equivalent to the LCS. The recovery is to be within the current water LCS QC limits for the known concentrations and 30% RSD for all replicates.

18.2.2. Analysis of four (4) replicates of Ottawa sand spiked with 250µL of 8270C LCS Spiking Solution and 10µL of nNPDA plus all other compounds that are currently reported at a concentration of 1670µg/kg or equivalent to the LCS. The recovery is to be within the current LCS QC acceptance limits for the known concentration and 30% RSD for all replicates.

19. Method Modifications

19.1. Method modifications for EPA method 8270C are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- All major modification to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- The laboratory follows the DFTPP Tune criteria outlined in EPA 525.2.
- The laboratory practice is to have thermal preservation at $\leq 6^{\circ}\text{C}$. This is based on 40CFR Part 136, page 29808, footnote 18.
- If a client fails to provide the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument operations manual or the SOP S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance* (current revision or replacement).

21. Troubleshooting

21.1. Please refer to the instrument manufacturer operations manual.

22. Safety

22.1. **Standards and Reagents:** The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples:** Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. **Equipment:** Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The GC pneumatic system uses gas under high pressure. This high pressure introduces the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

- 25.1. USEPA, SW-846, Method 8270C, “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.
- 25.2. USEPA, SW-846, Method 8000B, “Determinative Chromatographic Separations”, December 1996.
- 25.3. USEPA, Method 625, Appendix A to Part 136, (1984), “Base/Neutrals and Acids”.
- 25.4. USEPA, Method 525.2, Revision 2.0 (1995), “Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry”.
- 25.5. Pace Quality Assurance Manual- most current version.
- 25.6. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, “Quality Systems”- most current version.
- 25.7. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

26. Tables, Diagrams, Flowcharts, and Validation Data

- 26.1. Attachment I: Client Specific Requirements.
- 26.2. Attachment II: Tailing Factor Calculation

27. Revisions

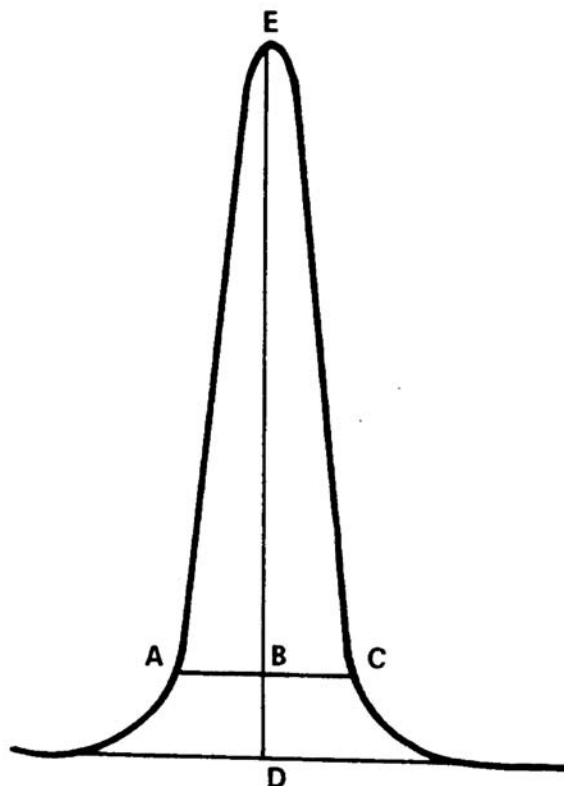
Document Number	Reason for Change	Date
S-GB-O-049-Rev.05	Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 <i>Preparation of SOPs</i> Throughout Document: Renamed Tables to be consistent with current Section. Section 11.3: Changed ICV calculation criteria to match CCV calculation criteria. Table 11.Section 12.2.1: Updated DFTPP Tune Criteria to be consistent with EPA 525.2. Section 12.3: Added tailing factor criteria. Section 19: Added Modification in relation to tune criteria. Attachment II: Tailing Factor Calculation added.	30May2013
S-GB-O-049-Rev.06	Throughout Document: Updated laboratory name to Pace Analytical Services LLC – Green Bay WI Table 7.1: Updated temperature to $\leq 6^{\circ}\text{C}$ from $4\pm 2^{\circ}\text{C}$. Table 9.1 and 12.1: Updated information for 40MSS6/40MSS8, added 40MSSA. Table 9.2: Updated with current vendor information. Section 10.1: Added Acetone. Table 10.4: Changed standard and solvent amounts in calibration curve. Table 11.1: Added pyridine. Table 11.2: Updated SOP reference to most current revision. Table 11.3.1: Added CRDL language. Table 12.2: Updated 1° and 2° ions. Table 13: Updated MB criteria from 20X to 10X rule for qualification requirements. Section 22.1: Updated MSDS to SDS Section 23.1: Updated SOP reference	24Oct2016
S-GB-O-049-Rev.07	Section 12.11.1: Added to section that data reduction must be done on every sample and quality control standard.:	21Jun2017

Attachment I:

Throughout document, reference to Client Specific requirements refers to samples analyzed following: BP Technical Requirements LaMP Revision 10.1, Canadian National Railway Services and Technical Specifications Manual, GE Minimum Standards Revision 2.

Attachment II: DFTPP Tailing Factor Calculation

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm

10% Peak Height = BD = 10 mm

Peak Width at 10% Peak Height = AC = 23 mm

AB = 11 mm

BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Figure 13. Tailing factor calculation.



Pace Analytical Services, LLC Green Bay, WI
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469-2436
Fax: 920 469-8827


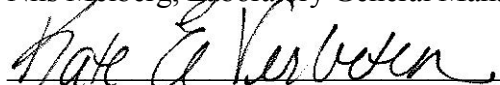
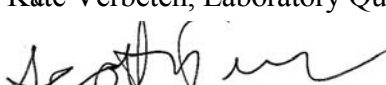
STANDARD OPERATING PROCEDURE

Determination of Volatile Organics by GC/MS

Reference Methods: EPA Method SW-846 8260B with 5030B and 5035; and
EPA Method 624

LOCAL SOP NUMBER:	S-GB-O-056-Rev.11
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-056-Rev.10
SOP TEMPLATE NUMBER:	TMP-ALL-O-002-Rev.01

LOCAL APPROVAL

 Nils Melberg, Laboratory General Manager	<u>06/21/17</u> Date
 Kate Verbeten, Laboratory Quality Manager	<u>6/21/17</u> Date
 Scott Turner, Department Manager	<u>6/21/2017</u> Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Green Bay to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed SW-846 Methods 5030B, 5035 and 8260B; and EPA 624.

2. Summary of Method

Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract by purging with helium or nitrogen. The purged analytes are collected in a trap. At the completion of the purge time, the trap is rapidly heated and back flushed with helium to drive out the trapped analytes. The analytes are transferred into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. The mass spectrometer is operated in a repetitive scan mode. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

3. Scope and Application

- 3.1. Personnel:** This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.
- 3.2. Parameters:** This SOP applies to compounds listed in Section 11, Table 11.1 Calibration standard compound concentrations, analyzed by SW-846 Methods 5030B, 5035 and 8260B and EPA 624. This method is applicable to most organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates). This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

4. Applicable Matrices

- 4.1.** This SOP is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data.

5. Limits of Detection and Quantitation

- 5.1.** The reporting limit (PQL) for all analytes can be found Section 11, Table 11.1. All current MDLs are listed in the LIMs and are available by request from the Quality Manager.

6. Interferences

- 6.1.** Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. The use of polytetrafluoroethylene (PTFE, Teflon) as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.
- 6.2.** A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action to this condition is rinsing the purging apparatus and sample syringes with two or more portions of organic free water between samples. Analyze one or more blanks to check for cross contamination prior to sample analysis.
- 6.3.** Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area should be located as far as possible from areas where these solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

7. Sample Collection, Preservation, Shipment and Storage

Table 7.1: Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Three (3) VOA vials	Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace <i>Note:</i> 2-CLEVE, Styrene, and Vinyl Chloride requires an unpreserved sample.	≤6°C	Unpreserved: 7 days pH Preserved: 14 days
Low Level Aliquot Soil/Solid (non-aqueous)	One (1) 2-4 oz. wide mouth jar for % moisture AND Two (2) 5-g aliquots in vials with magnetic stir bar, 5.0 mL reagent water and 1.0 g sodium bisulfate as needed. <i>OR (alternative):</i> Two (2) EnCore, TerraCore or similar sampling tubes.	No preservation <i>OR</i> sodium bisulfate Note: If sample effervesces on contact with the preservative, the sodium bisulfate should be eliminated for that sample.	With sodium bisulfate: ≤6°C Without preservation (including EnCore, TerraCore or similar): 4 ± 2°C for up to 48 hours before storing between -7°C and -20°C, inclusive, until analysis.	Unpreserved or not stored frozen: 48 hours Preserved with sodium bisulfate or stored frozen: 14 days
High Level Aliquot Soil/Solid (non-aqueous)	One (1) 10-g aliquot in vial with 10.0 mL purge and trap grade MeOH. <i>OR (alternative)</i> One (1) 10-g aliquot in empty vial	Methanol - if sample was collected in empty vial it must be transferred into 10 mL of purge & trap grade MeOH within 48 hours of collection	With methanol: ≤6°C.	Unpreserved: 48 hours Preserved with methanol: 14 days WI Only: 21 days
TCLP Leachates	Tedlar bag or THREE (3) VOA vials.	Filled and capped to eliminate any headspace. Vials with bubbles larger than 5 mm should be discarded.	≤6°C	14 days from end of leaching procedure

Table 7.2: Trip Blank Requirements

Aqueous	Low Level Aliquot Soil/Solid	High Level Aliquot Soil/Solid
One (1) 40mL VOA vial w/ reagent DI water	One (1) 40mL VOA vial w/ 5mL sodium bisulfate (or reagent DI water) and magnetic stir bar	One (1) 40mL VOA vial w/ 5mL purge and trap grade MeOH

8. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

- 8.1. Run Sequence Log** – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

8.2. Toxicity Characteristic Leaching Procedure (TCLP) – An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

8.3. Tune Period – The period after the BFB instrument tune check within which analyses may be performed.

9. Equipment and Supplies

Table 9.1: Equipment

Analytical Instrument/Peripherals	EPIC Pro Name
HP 5890 Series II GC	40MSV1
HP 5972 MSD	40MSV1
Archon Autosampler	40MSV1
Tekmar 3000 Purge and Trap Concentrator	40MSV1
HP 6890 GC	40MSV2
HP 5973 MSD	40MSV2
Archon Autosampler	40MSV2
Tekmar 3000 Purge and Trap Concentrator	40MSV2
Agilent 6850 GC	40MSV3
Agilent 5975 MSD	40MSV3
Tekmar Aquatek 70	40MSV3
Tekmar Stratum Purge and Trap Concentrator	40MSV3
HP 6890 GC	40MSV5
HP 5973 MSD	40MSV5
Archon Autosampler	40MSV5
Tekmar 3000 Purge and Trap Concentrator	40MSV5
HP 6890 GC	40MSV7
HP 5973 MSD	40MSV7
Archon Autosampler	40MSV7
Tekmar 3000 Purge and Trap Concentrator	40MSV7
Agilent Technologies 6850 Network GC System	40MSV8
Agilent Technologies 5975B MSD	40MSV8
EST 8100 Autosampler	40MSV8
Teledyne Tekmar 14-9800-100 Stratum Purge and Trap System	40MSV8
Agilent 5975C GCMS	40MSVA
Agilent 7890A GC	40MSVA
EST 8100 Autosampler	40MSVA
Tekmar Stratum Purge and Trap Concentrator	40MSVA
Agilent 5975C GCMS	40MSVB
Agilent 7890A GC	40MSVB
Tekmar Atomx Autosampler/Purge and Trap Conc.	40MSVB
Agilent 7890B GC	40MSVC
Agilent 5977A MSV	40MSVC
Tekmar Stratum Purge and Trap Concentrator	40MSVC
EST Centurion Autosampler	40MSVC

Table 9.2: Supplies

Supplies	Manufacturer	Vendor	Catalog #
10µL Gastight 1701	Hamilton	Fisher Scientific	14-815-1
25µL Gastight 1702	Hamilton	Fisher Scientific	14-815-29
50µL Gastight 1705	Hamilton	Fisher Scientific	14-824-30
100µL Gastight 1710	Hamilton	Fisher Scientific	13-684-100
250µL Gastight 1725	Hamilton	Fisher Scientific	13-684-102
500µL Gastight 1750	Hamilton	Fisher Scientific	13-684-106
1mL Gastight 1001	Hamilton	Fisher Scientific	14-824-25
5mL Gastight 1005	Hamilton	Fisher Scientific	13-684-96
50mL Gastight 1050	Hamilton	Fisher Scientific	14-815-195
DB-624 Capillary column, 20mX0.18 mm i.d.X1.0 µm	J&W Scientific	VWR Scientific	121-1324
K-Trap, Vocab3000, Tekmar3000	Supelco	Supelco	24920-U
Fritless 5 mL Sparge Tube	Supelco	Supelco	22780
IceBlue Septa, 11mm	Restek	Restek	22392
Single Gooseneck Injection port liners (4mm)	Restek	Restek	20799
Gold-plated inlet seals	Restek	Restek	21306
Viton O-rings	Restek	Restek	20377
0.4mm Vespel/Graphite ferrules	Restek	Restek	20211
GCMS Filaments	Agilent Technologies	Agilent Technologies	05972-60053
Stir Bar	Fisher Brand	Fisher Brand	14-511-60A
40 mL VOA vials	QEC	QEC	3112-40mL
10 mL volumetric	Kimax Brand	Fisher Scientific	10-212AA
25 mL volumetric	Kimax Brand	Fisher Scientific	10-212BB
50 mL volumetric	Kimax Brand	Fisher Scientific	10-212A
100 mL volumetric	Kimax Brand	Fisher Scientific	10-212B
200 mL volumetric	Kimax Brand	Fisher Scientific	10-212C
500 mL volumetric	Kimax Brand	Fisher Scientific	10-218D
Pasteur Pipettes	Fisher Scientific	Fisher Scientific	13-678-20A
Pipette bulb	Fisher Scientific	Fisher Scientific	14-065B
0.1-2.5 mL Repipettor	Brinkmann	Fisher Scientific	13-688-130
1-5 mL Repipettor	Brinkmann	Fisher Scientific	13-688-131
2-10 mL Repipettor	Brinkmann	Fisher Scientific	13-688-133
5-25 mL Repipettor	Brinkmann	Fisher Scientific	13-688-134
1.8 mL amber vials & caps	Restek	Restek	24637
10 mL graduate cylinder	Kimax Brand	Fisher Scientific	08-554B "to deliver"
40 mL VOA vials HCl preserved	QEC	QEC	3112-40HCl
2 oz. jars with Teflon lids	QEC	QEC	2114-0002
Spatulas	Fisher Scientific	Fisher Scientific	14-511-60A

10. Reagents and Standards

10.1. Reagents

Table 10.1: Reagents

Reagent	Conc.	Purity	Manufacturer	Vendor	Catalog #
Methanol	100%	Purge and Trap grade	Burdick & Jackson	VWR Scientific	232-1
Sodium Bisulfate	Granular	Certified grade	Fisher Scientific	Fisher Scientific	S-240-3
Nitrogen gas		99.999%	Michigan Airgas	Michigan Airgas	
Helium gas		99.999%	Michigan Airgas	Michigan Airgas	
Reverse Osmosis (ROW) Water		Organic Free	Flowmatic	Culligan	
Ottawa Sand, 20-30 mesh		ASTM C190	Fisher Scientific	Fisher Scientific	S23-3

10.2. Analytical Standards

10.2.1. Definitions: Standards are required for mass spectrometer tuning, initial calibration, calibration verification standards, second source verification, internal standards, surrogates, and for preparing LCS, MS, and MSD samples. Table 10.2 describes the standards used. Table 10.3 lists the stock standards used. Table 10.4 lists the compounds in each stock standard.

Table 10.2: Standard Definitions

Standard	Description	Comments
Tune Standard	4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis	Must purge between 5 and 50ng
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	For volatiles analysis, this may be used as the LCS if analyzed once every 20 samples.
Internal Standard	A solution added all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Pentafluorobenzene 1,4-Difluorobenzene Chlorobenzene-d5 1,4-Dichlorobenzene-d4
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to analysis.	Dibromofluoromethane Toluene-d8 4-Bromofluorobenzene
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	For volatiles analysis, this can be used as the second source verification standard.

10.2.2. Stock Standards

Table 10.3: Stock Standards

Standard	Conc.	Purity	Manufacturer	Vendor	Catalog #
4-BFB Tuning Standard	5000 µg/mL	99%	Restek	Restek	30003-520
502.2 Cal Gases Mix	2000 µg/mL	99%	Restek	Restek	30042
502.2 ICV Gases Mix	2000 µg/mL	99%	Accustandard	Accustandard	M-502B-10X
502.2 Cal 2000 Megamix	2000 µg/mL	99%	Restek	Restek	30431
Megamix – ICV	2000 µg/mL	99%	o2si	o2si	122708-05
Vinyl Acetate	Neat	99+%	Chem Service	Chem Service	F718
Calibration Ketone Mix	5000 µg/mL	99%	Restek	Restek	30006
Ketones – ICV	2000 µg/mL	99%	o2si	o2si	121020-10
Pace GB Custom Mix #3	various µg/mL	99+%	o2si	o2si	120407-11-SS
Custom – ICV	100,000 – 200,000 µg/µL	99%	o2si	o2si	122707-05
Acrolein – CAL	Neat	99+%	Chem Service	Chem Service	N-11030-1G
2-Chloroethylvinyl Ether	2000 µg/mL	99+%	Restek	Restek	30265
Reactive Mix – ICV	100-1000 µg/mL	99%	o2si	o2si	120407-13
8260 Internal Standard	2500 µg/mL	99%	Restek	Restek	30173
8260 Surrogate Standard	2500 µg/mL	99%	Restek	Restek	30174
CLP 4.1Mega Mix	2000 µg/mL	99%	Restek	Restek	30456
502.2 Gases Mix #1	2000 µg/mL	99%	Restek	Restek	30006

10.2.3. Standard Mixes

Table 10.4: Standard Mixes

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
Restek	4-BFB Tuning Standard	30003-520	4-bromofluorobenzene	2500
Restek	502.2 Cal Gases Mix	30042	Bromomethane	2000
			Chloroethane	2000
			Chloromethane	2000
			Dichlorodifluoromethane	2000
			Trichlorofluoromethane	2000
			Vinyl Chloride	2000
Accustandard	502.2 ICV Gases Mix =	M-502B-10X	Bromomethane	2000
			Chloroethane	2000
			Chloromethane	2000
			Dichlorodifluoromethane	2000
			Trichlorofluoromethane	2000
			Vinyl Chloride	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
Restek	502.2 Cal 2000 MegaMix	30431	1,1,1,2-Tetrachloroethane	2000
			1,1,1-Trichloroethane	2000
			1,1,2,2-Tetrachloroethane	2000
			1,1,2-Trichloroethane	2000
			1,1-Dichloroethane	2000
			1,1-Dichloropropene	2000
			1,1-Dichloroethene	2000
			1,2,3-Trichlorobenzene	2000
			1,2,3-Trichloropropane	2000
			1,2,3-Trimethylbenzene	2000
			1,2,4-Trimethylbenzene	2000
			1,2-Dibromo-3-chloropropane	2000
			1,2-Dibromoethane	2000
			1,2-Dichloroethane	2000
			1,2-Dichlorobenzene	2000
			1,2-Dichloropropane	2000
			1,3,5-Trimethylbenzene	2000
			1,3-Dichlorobenzene	2000
			1,3-Dichloropropane	2000
			1,4-Dichlorobenzene	2000
			2,2-Dichloropropane	2000
			2-Chlorotoluene	2000
			4-Chlorotoluene	2000
			4-Isopropyltoluene	2000
			Benzene	2000
			Bromobenzene	2000
			Bromochloromethane	2000
			Bromodichloromethane	2000
			Bromoform	2000
			Carbon Tetrachloride	2000
			Chlorobenzene	2000
			Chloroform	2000
			cis-1,2-Dichloroethene	2000
			cis-1,3-Dichloropropene	2000
			Dibromochloromethane	2000
			Dibromomethane	2000
			Ethylbenzene	2000
			Hexachloro-1,3-butadiene	2000
			Isopropylbenzene	2000
			Methylene Chloride	2000
			m-Xylene	2000
			Naphthalene	2000
			n-Butylbenzene	2000
			n-Propylbenzene	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
			o-Xylene	2000
			p-Xylene	2000
			sec-Butylbenzene	2000
			Styrene	2000
			tert-Butylbenzene	2000
			Tetrachloroethene	2000
			Toluene	2000
			trans-1,2-Dichloroethene	2000
			trans-1,3-Dichloropropene	2000
			Trichloroethene	2000
o2si	MegaMix – ICV	122708-05	Isopropyl alcohol	20,000
			Isobutyl alcohol	20,000
			tert-butyl alcohol	10,000
			Acetonitrile	5000
			Ethyl ether	2000
			Isopropyl ether	2000
			Benzene	2000
			n-propylbenzene	2000
			sec-butylbenzene	2000
			tert-butylbenzene	2000
			1,2,4 trimethylbenzene	2000
			n-butylbenzene	2000
			naphthalene	2000
			p-cymene	2000
			1,2-dichlorobenzene	2000
			1,3-dichlorobenzene	2000
			Chlorobenzene	2000
			1,2,3-trichlorobenzene	2000
			1,2,4-trichlorobenzene	2000
			Bromobenzene	2000
			Bromochloromethane	2000
			Carbon tetrachloride	2000
			Dibromomethane	2000
			Bromodichloromethane	2000
			Bromoform	2000
			Dibromochloromethane	2000
			trans-1,2-dichloroethylene	2000
			1,1-dichloroethylene	2000
			1,1-dichloroethane	2000
			1,1,1-trichloroethane	2000
			2,2-dichloropropane	2000
			tetrachloroethylene	2000
			1,1,1,2-tetrachloroethane	2000
			1,1,2-trichloroethane	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
			1,2-dichloroethane	2000
			1,2-dibromo-3chloropropane	2000
			1,2-dibromomethane	2000
			1,1 dichloropropylene	2000
			1,2,3-trichloropropane	2000
			1,2 dichloropropane	2000
			trans-1,3-dichloropropylene	2000
			cis-1,3-dichloropropylene	2000
			1,3-dichloropropane	2000
			Iodomethane	2000
			Carbon disulfide	2000
			Methyl acetate	2000
			Cyclohexane	2000
			Methyl t-butyl ether	2000
			Ethyl t-butyl ether	2000
			tert-amyl methyl ether	2000
			1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	2000
			Heptane (C7)	2000
			1,2,3-trimethylbenzene	2000
			n-hexane (C6)	2000
			Isopropyl acetate	2000
			1-methylnaphthalene	2000
			2-methylnaphthalene	2000
			Acrylonitrile	2000
			Allyl chloride	2000
			Chloroform	2000
			2-chlorotoluene	2000
			4-chlorotoluene	2000
			Cis-1,2-dichloroethylene	2000
			1,4-dichlorobenzene	2000
			Cis-1,4-dichloro-2-butene	2000
			2,3-dichloro-1-propene	2000
			Ethylbenzene	2000
			Hexachlorobutadiene	2000
			Hexachloroethane	2000
			Isopropylbenzene	2000
			Methyl cyclohexane	2000
			Methylene chloride	2000
			Styrene	2000
			1,1,2,2-tetrachloroethane	2000
			Tetrahydrofuran	2000
			Toluene	2000
			Trichloroethylene	2000
			1,3,5-Trimethylbenzene	2000
			m-xylene	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
			o-xylene	2000
			p-xylene	2000
Chem Service	Vinyl Acetate	F718	Vinyl Acetate	Neat
Restek	Calibration Ketone Mix	30006	2-Butanone	5000
			2-Hexanone	5000
			4-Methyl-2-pentanone	5000
			Acetone	5000
o2si	Ketones – ICV	121020-10	2-Butanone	2000
			2-Hexanone	2000
			4-Methyl-2-pentanone	2000
			Acetone	2000
o2si	Pace-GB Custom	121082-01-SS	Dichlorofluoromethane	2000
	Mix #3		1,1,2-trichlorotrifluoroethane	2000
			Acetonitrile	5000
			Iodomethane (methyl iodide)	2000
			Allyl chloride (3-chloropropene)	2000
			Carbon disulfide	2000
			Acrylonitrile	2000
			2,3-dichloropropylene	2000
			cis-1,4-dichloro-2-butene	2000
			trans-1,4-dichloro-2-butene	2000
			1,2,3-trimethylbenzene	2000
			Hexachloroethane	2000
			2-methylnaphthalene	2000
			1-methylnaphthalene	2000
			Ethanol	100,000
			Diethyl ether (ethyl ether	2000
			2-propanol (isopropanol)	20,000
			tert-butanol (TBA)	10,000
			Methyl acetate	2000
			n-Hexane	2000
			Methyl-tert-butyl-ether (MTBE)	2000
			1-Propanol	100,000
			1,4-dioxane	100,000
			1-butanol	100,000
			Tetrahydrofuran	2000
			Cyclohexane	2000
			Heptane (C7)	2000
			tert-amyl methyl ether	2000
			Ethyl t-butyl ether	2000
			Isopropyl ether	2000
			Isobutyl alcohol	20,000

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
			Methyl cyclohexane	2000
			Isopropyl acetate	2000
Chem Service	Acrolein - Cal	N-11030-1G	Acrolein	Neat
Restek	2-chloroethylvinyl ether	30265	2-chloroethylvinyl ether	2000
o2si	Reactive Mix – ICV	120407-13	2-chloroethylvinyl ether	100
			Acrolein	1000
			Vinyl Acetate	100
Restek	8260 Internal Standard	30173	Pentafluorobenzene	2500
			1,4-Difluorobenzene	2500
			Chlorobenzene-d5	2500
			1,4-Dichlorobenzene-d4	2500
Restek	8260 Surrogate Standard	30174	Dibromofluoromethane	2500
			Toluene-d8	2500
			1-Bromo-4-fluorobenzene	2500
Restek	CLP 4.1Mega Mix	30456	1,1,2-trichlorotrifluoroethane	2000
			1,1-dichloroethene	2000
			Benzene	2000
			Bromodichloromethane	2000
			Bromoform	2000
			Carbon Tetrachloride	2000
			Chlorobenzene	2000
			Chloroform	2000
			Dibromochloromethane	2000
			1,2-dichloroethane	2000
			Cis-1,2-dichloroethene	2000
			1,2-Dichloropropane	2000
			trans-1,3-dichloropropylene	2000
			cis-1,3-dichloropropylene	2000
			Ethylbenzene	2000
			Styrene	2000
			1,1,2,2-tetrachloroethane	2000
			Tetrachloroethene	2000
			Toluene	2000
			1,1,1-trichloroethane	2000
			1,1,2-trichloroethane	2000
			Trichloroethane	2000
			m&p-Xylene	4000
			o-Xylene	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (µg/mL)
			Cyclohexane	2000
			Methylcyclohexane	2000
			1,2-dibromo-3chloropropane	2000
			1,2-Dibromoethane	2000
			Isopropylbenzene	2000
			1,2,4-Trichlorobenzene	2000
			1,3-Dichlorobenzene	2000
			1,4-Dichlorobenzene	2000
			1,2-Dichlorobenzene	2000
			Methyl acetate	2000
			Carbon disulfide	2000
			Methylene chloride (dichloromethane)	2000
			Methyl-tert-butyl ether (MTBE)	2000
			Trans-1,2-dichloroethene	2000
			1,1-dichloroethane	2000

10.2.4. Standard Storage Conditions

Table 10.5: Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	Concentrated reference solution purchased directly from approved vendor	<ol style="list-style-type: none"> 1. Manufacturer's recommended expiration date for unopened ampulated standards. 2. Gas standards must be replaced 6 months after ampule is opened. 3. All other stock standards must be replaced 6 months after ampule is opened or on expiration date, whichever is sooner. 	<ol style="list-style-type: none"> 1. Manufacturer's recommended storage conditions 2. When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	Reference solutions prepared by dilutions of the stock solution	<ol style="list-style-type: none"> 1. 6 months from preparation or the expiration date listed for the stock source, whichever is sooner. 2. 6 months for gas working standards. 3. Working solutions must be checked frequently and replaced if degradation or evaporation is suspected. 	<ol style="list-style-type: none"> 1. Store in amber vials with Teflon lined screw caps 2. Manufacturer's recommended storage conditions for stock source solution. 3. If stock source conditions conflict, store standard at coldest condition of any source.

10.2.5. Standard Sources

Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials are listed in Table 10.3. The recipes for preparing dilutions and all working and intermediate standards, and concentrations for all compounds are presented in Tables 10.6 and 10.7. All intermediate standards are prepared using purge and trap grade methanol and stored frozen in glass vials with Teflon lined screw caps or Mininert valves or as recommended by the standard manufacturer.

10.2.6. Preparation Procedures

Table 10.6: Intermediate Standard Preparation

Standard	Acronym	Concentration	Direction found in Section:
Level 1 Calibration Standard (MeOH Curve Only)	CAL1 (MeOH Curve Only)	0.40µg/L all compounds except as follows: 1.0 µg/L acetonitrile; 2.0 µg/L tert butyl alcohol; 4.0 µg/L acrolein, isopropanol, isobutanol; 20 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	Table 10.7
Water Level 1 Calibration Standard	CAL1 (Water)	1.0µg/L all compounds except as follows: 2.5 µg/L acetonitrile; 5.0 µg/L tert butyl alcohol; 10 µg/L acrolein, isopropanol, isobutanol;	Table 10.7
MeOH Level 2 Calibration Standard	CAL2 (MeOH)	50 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	
Water Level 2 Calibration Standard	CAL2 (Water)	5.0 µg/L all compounds except as follows: 12.5 µg/L acetonitrile; 25 µg/L tert butyl alcohol; 50 µg/L acrolein, isopropanol, isobutanol;	Table 10.7
MeOH Level 3 Calibration Standard	CAL3 (MeOH)	250 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	
Water Level 3 Calibration Standard	CAL3 (Water)	20 µg/L all compounds except as follows: 50 µg/L acetonitrile,; 100 µg/L tert butyl alcohol; 200 µg/L acrolein, isopropanol, isobutanol;	Table 10.7
MeOH Level 4 Calibration Standard	CAL4 (MeOH)	1000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	
Water Level 4 Calibration Standard	CAL4 (Water)	50 µg/L all compounds except as follows: 125 µg/L acetonitrile; 250 µg/L tert butyl alcohol;	Table 10.7
MeOH Level 5 Calibration Standard	CAL5 (MeOH)	500 µg/L acrolein, isopropanol, isobutanol; 2500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	
Water Level 5 Calibration Standard	CAL5 (Water)	100 µg/L all compounds except as follows: 250 µg/L acetonitrile; 500 µg/L tert butyl alcohol; 1000 µg/L acrolein, isopropanol, isobutanol;	Table 10.7
MeOH Level 6 Calibration Standard	CAL6 (MeOH)	5000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	
Water Level 6 Calibration Standard	CAL6 (Water)	200 µg/L all compounds except as follows: 500 µg/L acetonitrile; 1000 µg/L tert butyl alcohol; 2000 µg/L acrolein, isopropanol, isobutanol;	Table 10.7
MeOH Level 7 Calibration Standard	CAL7 (MeOH)	10000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	
Level 7 Calibration Standard (Water Curve only)	CAL7 (Water Curve only)	300 µg/L all compounds except as follows: 750 µg/L acetonitrile; 1500 µg/L tert butyl alcohol; 3000 µg/L acrolein, isopropanol, isobutanol; 15000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Table 10.7
*Optional Water Level 3 Calibration	CAL3 (Water)	10 µg/L all compounds except as follows:	Table 10.7

Standard	Acronym	Concentration	Direction found in Section:
Standard * If this standard is made for a water Curve, all subsequent Water Level # will increment by 1 (i.e. 300µg/L will now be CAL8)	Curve only) *Optional	25 µg/L acetonitrile; 50 µg/L tert butyl alcohol; 100 µg/L acrolein, isopropanol, isobutanol; 500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	
Independent Calibration Verification Standard	ICV050	50 µg/L all compounds except as follows: 125 µg/L acetonitrile; 250 µg/L tert butyl alcohol; 500 µg/L acrolein, isopropanol, isobutanol; 2500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Table 10.7
Calibration Verification Standard	CCV050	50 µg/L all compounds except as follows: 125 µg/L acetonitrile; 250 µg/L tert butyl alcohol; 500 µg/L acrolein, isopropanol, isobutanol; 2500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Table 10.7
Acrolein – Cal Intermediate	Acrolein - Cal	20,000 µg/mL	Table 10.7
Vinyl Acetate – Cal Intermediate	Vinyl Acetate - Cal	10,500 µg/mL	Table 10.7
Surrogate Standard	SS	50 µg/L	Table 10.7
Internal Standard	IS	50 µg/L	Table 10.7
Method Blank	MB	< Reporting Limit	Table 10.7
BFB	TUNExxx	50 ng injection	Table 10.7
Matrix Spike/ Lab Control Spike Stock Solution - CLP 4.1 List for Water and Low Level Soil Samples	MS/LCS CLP 4.1 List Stock Solution for Water and Low Level Soil Samples	100 µg/mL for all compounds	Table 10.7
Matrix Spike/Lab Control Spike Stock Solution - CLP 4.1 List for Methanol Preserved Soil Samples	MS/LCS CLP 4.1 List Stock Solution for Methanol Preserved Soil Samples	500 µg/mL for all compounds	Table 10.7
Matrix Spike/Lab Control Spike Stock Solution - Full List for Water and Low Level Soil Samples	MS/LCS Stock Solution – Full List for Water and Low Level Soil Samples	100 µg/mL all compounds except as follows: 1000 µg/mL Acrolein	Table 10.7
Matrix Spike/ Lab Control Spike Stock Solution - Full List for Methanol Preserved Soil Samples	MS/LCS Stock Solution – Full List for Methanol Preserved Soil Samples	500 µg/mL all compounds except as follows: 5000 µg/mL Acrolein	Table 10.7
MS/MSD – LCS/LCSD Spike for Water	MS/MSD	50 µg/L for most compounds, Various for	Table 10.7

Standard	Acronym	Concentration	Direction found in Section:
Samples	LCS/LCSD	others.	
MS/MSD – LCS/LCSD Spike for 624 Water Samples	MS/MSD LCS/LCSD	20 µg/L for most compounds, Various for others.	Table 10.7
MS/MSD – LCS/LCSD Spike for Low Level Soil Samples	MS/MSD LCS/LCSD	50 µg/kg for most compounds, Various for others.	Table 10.7
MS/MSD – LCS/LCSD Spike for Methanol Preserved Soil Samples	MS/MSD LCS/LCSD	2500 µg/kg for most compounds, Various for others.	Table 10.7
Extraction Blank	EBLK	< Reporting Limit	

Table 10.7: Preparation of Standards

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
4-Bromofluorobenzene	BFB	50 µg/mL	100µL of 5000 µg/mL BFB into methanol	10 mL
Internal Standard	IS	250 µg/mL	5000 µL of 2500 µg/mL IS into methanol	50 mL
Surrogate Standard	SS	250 µg/mL	5000 µL of 2500 µg/mL SS into methanol	50 mL
Internal/Surrogate Std.	IS/SS	250 µg/mL	5000 µL of 2500 µg/mL IS/SS into MeOH	50 mL
Vinyl Acetate – Cal Intermediate	Vinyl Acetate - Cal	10,500 µg/mL	0.105g of neat Vinyl Acetate Diluted into methanol	10 mL
Acrolein – Cal Intermediate	Acrolein - Cal	20,000 µg/mL	0.21g of neat Acrolein 1 mL Reverse Osmosis Water Diluted into methanol	10 mL
100 µg/mL Calibration Std. 250 µg/mL acetonitrile; 500 µg/mL tert butyl alcohol; 1000 µg/mL acrolein, isopropanol, isobutanol; 5000 µg/mL 1,4-dioxane, 1-propanol, n-butanol, ethanol	CAL Stock Standard	100 µg/mL all compounds except as follows: 250 µg/mL acetonitrile,; 500 µg/mL tert butyl alcohol; 1000 µg/mL acrolein, isopropanol, isobutanol; 5000 µg/mL 1,4-dioxane, 1-propanol, n-butanol, ethanol	1250 µL of 2000 µg/mL 502.2 Calibration Gases Mix 1250 µL of 2000 µg/mL 502.2 Cal 2000 Megamix 238 µL of 10,500 µg/mL Vinyl Acetate -Cal 500 µL of 5000 µg/mL Calibration KetoneMix 1250 µL of 2000 µg/mL Pace GB Custom Mix #3 1250 µL of 20000 µg/mL Acrolein -Cal 1250 µL of 2000 µg/mL 2-Chloroethylvinyl ether Diluted into methanol Surrogates are added by the instrument during aqueous and low level soil calibration events.	25 mL
100 µg/mL Calibration Std. 250 µg/mL acetonitrile; 500 µg/mL tert butyl alcohol; 1000 µg/mL acrolein, isopropanol, isobutanol; 5000 µg/mL 1,4-dioxane, 1-propanol, n-butanol, ethanol	ICV Stock Standard	100 µg/mL all compounds except as follows: 250 µg/mL acetonitrile,; 500 µg/mL tert butyl alcohol; 1000 µg/mL, isopropanol, isobutanol; 5000 µg/mL 1,4-dioxane, 1-propanol, n-butanol, ethanol	1250 µL of 2000 µg/mL 502.2 ICV Gases Mix 1250 µL of 2000 µg/mL Megamix – ICV 1250 µL of 2000 µg/mL Ketones ICV 1250 µL of 2000 µg/mL Custom – ICV Diluted into methanol	25 mL
MeOH Level 1 Calibration Standard (MeOH Curve Only)	CAL1 (MeOH Curve Only)	0.40µg/L all compounds except as follows: 1.0 µg/L acetonitrile; 2.0 µg/L tert butyl alcohol; 4.0 µg/L acrolein, isopropanol, isobutanol; 20 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	Methanol curve only dilute: 2.0 µL 100 µg/mL CAL Stock Standard and 1 µL 250 µg/mL Surrogate Standard into 490 mL reverse osmosis water and 10 mL methanol	500 mL
Water Level 1 Calibration Standard	CAL1 (Water)	1.0µg/L all compounds except as follows: 2.5 µg/L acetonitrile; 5.0 µg/L tert butyl alcohol; 10 µg/L acrolein, isopropanol, isobutanol; 50 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	Water Curve: Dilute 5 µL of 100 µg/mL CAL Stock Standard into 500 mL reverse osmosis water.	500 mL
MeOH Level 2 Calibration Standard	CAL2 (MeOH)		If for a methanol curve dilute: 5 µL 100 µg/mL CAL Stock Standard and 2 µL 250 µg/mL Surrogate Standard into 490 mL reverse osmosis water and 10 mL methanol.	
Water Level 2	CAL2	5.0 µg/L all compounds	Water Curve:	50 mL

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
Calibration Standard MeOH Level 3 Calibration Standard	(Water) CAL3 (MeOH)	except as follows: 12.5 µg/L acetonitrile; 25 µg/L tert butyl alcohol; 50 µg/L acrolein, isopropanol, isobutanol; 250 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Dilute 2.5 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water. If for a methanol curve dilute: 2.5 µL 100 µg/mL CAL Stock Standard and 1 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 1 mL methanol.	
Water Level 3 Calibration Standard MeOH Level 4 Calibration Standard	CAL3 (Water) CAL4 (MeOH)	20 µg/L all compounds except as follows: 50 µg/L acetonitrile; 100 µg/L tert butyl alcohol; 200 µg/L acrolein, isopropanol, isobutanol; 1000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Water Curve: Dilute 10 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water. If for a methanol curve dilute: 10 µL 100 µg/mL CAL Stock Standard and 4 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 990 µL methanol.	50 mL
Water Level 4 Calibration Standard MeOH Level 5 Calibration Standard	CAL4 (Water) CAL5 (MeOH)	50 µg/L all compounds except as follows: 125 µg/L acetonitrile; 250 µg/L tert butyl alcohol; 500 µg/L acrolein, isopropanol, isobutanol; 2500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Water Curve: Dilute 25 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water. If for a methanol curve dilute: 25 µL 100 µg/mL CAL Stock Standard and 10 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 960 µL methanol.	50 mL
Water Level 5 Calibration Standard MeOH Level 6 Calibration Standard	CAL5 (Water) CAL6 (MeOH)	100 µg/L all compounds except as follows: 250 µg/L acetonitrile; 500 µg/L tert butyl alcohol; 1000 µg/L acrolein, isopropanol, isobutanol; 5000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Water Curve: Dilute 50 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water. If for a methanol curve dilute: 50 µL 100 µg/mL CAL Stock Standard and 20 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 930 µL methanol.	50 mL
Water Level 6 Calibration Standard MeOH Level 7 Calibration Standard	CAL6 (Water) CAL7 (MeOH)	200 µg/L all compounds except as follows: 500 µg/L acetonitrile; 1000 µg/L tert butyl alcohol; 2000 µg/L acrolein, isopropanol, isobutanol; 10000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Water Curve: Dilute 100 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water. If for a methanol curve dilute: 100 µL 100 µg/mL CAL Stock Standard and 40 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 860 µL methanol.	50 mL
Water Level 7 Calibration Standard (Water Curve only)	CAL7 (Water Curve only)	300 µg/L all compounds except as follows: 750 µg/L acetonitrile; 1500 µg/L tert butyl alcohol; 3000 µg/L acrolein, isopropanol, isobutanol; 15000 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Water Curve Only: Dilute 150 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water.	50 mL

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
*Optional Water Level3 Calibration Standard * If this standard is made for a water Curve, all subsequent Water Level # will increment by 1 (i.e. 300µg/L will now be CAL8)	CAL3 (Water Curve only) *Optional	10 µg/L all compounds except as follows: 25 µg/L acetonitrile; 50 µg/L tert butyl alcohol; 100 µg/L acrolein, isopropanol, isobutanol; 500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol.	Water Curve: Dilute 5 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water.	50 mL
Independent Calibration Verification Standard	ICV050	50 µg/L all compounds except as follows: 125 µg/L acetonitrile; 250 µg/L tert butyl alcohol; 500 µg/L acrolein, isopropanol, isobutanol; 2500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	Water Curve: Dilute 25 µL of 100 µg/mL ICV Stock Standard and 25 µL Reactive Mix – ICV into 50 mL reverse osmosis water. If for a methanol curve dilute: 25 µL 100 µg/mL ICV Stock Standard, 25 µL Reactive Mix – ICV and 10 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 940 µL methanol.	50 mL
Calibration Verification Standard	CCV050	50 µg/L all compounds except as follows: 125 µg/L acetonitrile; 250 µg/L tert butyl alcohol; 500 µg/L acrolein, isopropanol, isobutanol; 2500 µg/L 1,4-dioxane, 1-propanol, n-butanol, ethanol	Water Curve: Dilute 25 µL of 100 µg/mL CAL Stock Standard into 50 mL reverse osmosis water. If for a methanol curve dilute: 25 µL 100 µg/mL CAL Stock Standard and 10 µL 250 µg/mL Surrogate Standard into 49 mL reverse osmosis water and 960 µL methanol.	50 mL
Matrix Spike/Lab Control Spike Stock Solution - TCL 4.1 List for Water and Low Level Soil Samples	MS/LCS TCL 4.1 List Stock Solution for Water and Low Level Soil Samples	100 µg/mL for all compounds	1250 µL of 2000 µg/mL CLP 4.1Mega Mix 1250 µL of 2000 µg/mL 502.2 Gases Mix #1 Dilute into methanol.	25 mL
Matrix Spike/Lab Control Spike Stock Solution - TCL 4.1 List for Methanol Preserved Soil Samples	MS/LCS TCL 4.1 List Stock Solution for Methanol Preserved Soil Samples	500 µg/mL for all compounds	2500 µL of 2000 µg/mL CLP 4.1Mega Mix 2500 µL of 2000 µg/mL 502.2 Gases Mix #1 Dilute into methanol.	10 mL
Matrix Spike/Lab Control Spike Stock Solution - Full List *	MS/LCS Stock Solution – Full List	100 µg/mL all compounds except as follows: 250 µg/mL acetonitrile; 500 µg/mL tert butyl alcohol; 1000 µg/mL	1250 µL of 2000 µg/mL 502.2 ICV Gases Mix 1250 µL of 2000 µg/mL Megamix – ICV 1250 µL of 2000 µg/mL Ketones ICV 1250 µL of 2000 µg/mL Custom – ICV Diluted into methanol	25 mL

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
		isopropanol, isobutanol; 5000 µg/mL 1,4-dioxane, 1-propanol, n-butanol, ethanol		
MS/MSD – LCS/LCSD Spike for Water Samples	MS/MSD LCS/LCSD	50 µg/L for most compounds, Various for others.	Add 21 µL of the appropriate Matrix Spike/ Lab Control Spike Solution to 42 mL of sample for MS/MSD, or 42 mL of reagent water for LCS/LCSD	42 mL
MS/MSD – LCS/LCSD Spike for 624 Water Samples	MS/MSD LCS/LCSD	20 µg/L for most compounds, Various for others	Add 8.4 µL of the appropriate Matrix Spike / Lab Control Spike Solution to 42 mL of sample for MS/MSD or 42 mL of reagent water for LCS/LCSD.	42 mL
MS/MSD – LCS/LCSD Spike for Low Level Soil Samples	MS/MSD LCS/LCSD	50 µg/kg for most compounds, Various for others.	Add 2.5 µL of the appropriate Matrix Spike/ Lab Control Spike Solution to 5.0 g of sample containing 5 mL of reagent water for MS/MSD, or 5.0 g of Ottawa sand containing 5 mL of reagent water for LCS/LCSD	5 mL
MS/MSD – LCS/LCSD Spike for Methanol Preserved Soil Samples Standard List	MS/MSD LCS/LCSD	2500 µg/kg for most compounds, Various for others.	Add 50 µL of the Standard List Matrix Spike/ Lab Control Spike Solution to 10 g of sample containing 10 mL of methanol for MS/MSD, amount of Spike Solution is adjusted according to the initial methanol volume; or 10 g of Ottawa sand containing 10 mL of methanol for LCS/LCSD	10 mL
MS/MSD – LCS/LCSD Spike for Methanol Preserved Soil Samples Full List	MS/MSD LCS/LCSD	2500 µg/kg for most compounds, Various for others.	Add 250 µL of the Full List Matrix Spike/ Lab Control Spike Solution to 10 g of sample containing 10 mL of methanol for MS/MSD, amount of Spike Solution is adjusted according to the initial methanol volume; or 10 g of Ottawa sand containing 10 mL of methanol for LCS/LCSD	10 mL

*Samples to be analyzed by EPA 624 originating in the State of South Carolina must contain all analytes of interest in the LCS, MS, and MSD.

11. Calibration and Standardization

11.1. Tune Verification

The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method (see section 11.2), follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures.

11.2. Initial Calibration (ICAL)

11.2.1. Analysis of Standards

An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 11.1 for compound concentrations.

An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control

in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 12.6.3. Results for these TICs should be reported only on a present/absent basis. However, quantitative results may be reported provided they are qualified as estimated values.

Table 11.1: Calibration standard compound concentrations

Analyte	PQL water µg/L	PQL soil µg/kg	PQL 5035 soil µg/Kg	Cal1 MeOH curve only µg/L	Cal2 MeOH Cal1 water µg/L	Cal3 MeOH Cal2 water µg/L	Cal4 MeOH Cal3 water µg/L	Cal5 MeOH Cal4 water µg/L	Cal6 MeOH Cal5w ater µg/L	Cal7 MeOH Cal6 water µg/L	Cal7 water Curve only µg/L	* Optional Cal3 water Curve only µg/L
1,1-Dichloroethane	1	50	5	-	1	5	20	50	100	200	300	10
1,1-Dichloroethene	1	50	5	-	1	5	20	50	100	200	300	10
1,1-Dichloropropene	1	50	5	-	1	5	20	50	100	200	300	10
1,1,1-Trichloroethane	1	50	5	-	1	5	20	50	100	200	300	10
1,1,2-Trichloroethane	1	50	5	-	1	5	20	50	100	200	300	10
1,1,2-Trichloro- 1,1,2trifluoethane	5	50	5	-	1	5	20	50	100	200	300	10
1,1,1,2-Tetrachloroethane	1	50	5	-	1	5	20	50	100	200	300	10
1,1,2,2-Tetrachloroethane	1	50	5	-	1	5	20	50	100	200	300	10
1,2,4-Trichlorobenzene	5	250	5	-	1	5	20	50	100	200	300	10
1,2-Dichlorobenzene	1	50	5	-	1	5	20	50	100	200	300	10
1,2-Dibromo-3- chloropropane	5	250	5	-	1	5	20	50	100	200	300	10
1,2-Dichloroethane	1	50	5	-	1	5	20	50	100	200	300	10
1,2-Dibromoethane	1	50	5	-	1	5	20	50	100	200	300	10
1,2-Dichloropropane	1	50	5	-	1	5	20	50	100	200	300	10
1,2-Dichloroethene (Total)	2	100	10	-	2	10	40	100	200	400	600	20
1,2,3-Trimethylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
1,2,4-Trimethylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
1,2,3-Trichlorobenzene	5	50	5	-	1	5	20	50	100	200	300	10
1,2,3-Trichloropropane	1	50	5	-	1	5	20	50	100	200	300	10
1,3-Dichlorobenzene	1	50	5	-	1	5	20	50	100	200	300	10
1,3-Dichloropropane	1	50	5	-	1	5	20	50	100	200	300	10
1,3,5-Trimethylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
1,4-Dichlorobenzene	1	50	5	-	1	5	20	50	100	200	300	10
1,4-Dioxane (p-Dioxane)	250	12500	250	-	50	250	1000	2500	5000	10000	15000	500
1-Methylnaphthalene	5	250	5	-	1	5	20	50	100	200	300	10
2,2-Dichloropropane	1	50	5	-	1	5	20	50	100	200	300	10
2,3-Dichloropropene	1	50	5	-	1	5	20	50	100	200	300	10
2-Butanone (MEK)	20	250	20	-	1	5	20	50	100	200	300	10
2-Chlorotoluene	1	50	5	-	1	5	20	50	100	200	300	10
2-Chloroethylvinyl ether	5	250	10	-	1	5	20	50	100	200	300	10
2-Hexanone	5	250	5	-	1	5	20	50	100	200	300	10
2-Methylnaphthalene	5	250	5	-	1	5	20	50	100	200	300	10
2-Propanol	250	12500	50	-	10	50	200	500	1000	2000	3000	100
Allyl chloride	5	250	5	-	1	5	20	50	100	200	300	10
4-Chlorotoluene	1	50	5	-	1	5	20	50	100	200	300	10
TOTAL BTEX	1	300	30	-	1	5	20	50	100	200	300	10
Carbon disulfide	5	50	5	-	1	5	20	50	100	200	300	10
Ethanol	500	NA	500	-	50	250	1000	2500	5000	10000	15000	500
Acetone	20	250	20	-	1	5	20	50	100	200	300	10
Acrolein	20	2500	50	-	10	50	200	500	1000	2000	3000	100
Acetonitrile	20	250	12.5	-	2.5	12.5	50	125	250	500	750	25
Acrylonitrile	5	250	5	-	1	5	20	50	100	200	300	10

Analyte	PQL water µg/L	PQL soil µg/kg	PQL 5035 soil µg/Kg	Cal1 MeOH curve only µg/L	Cal2 MeOH Cal1 water µg/L	Cal3 MeOH Cal2 water µg/L	Cal4 MeOH Cal3 water µg/L	Cal5 MeOH Cal4 water µg/L	Cal6 MeOH Cal5w ater µg/L	Cal7 MeOH Cal6 water µg/L	Cal7 water Curve only µg/L	* Optional Cal3 water Curve only µg/L
Bromochloromethane	1	50	5	-	1	5	20	50	100	200	300	10
Benzene	1	20	5	0.4	1	5	20	50	100	200	300	10
Bromobenzene	1	50	5	-	1	5	20	50	100	200	300	10
Bromodichloromethane	1	50	5	-	1	5	20	50	100	200	300	10
Bromomethane	5	250	10	-	1	5	20	50	100	200	300	10
Bromoform	1	50	5	-	1	5	20	50	100	200	300	10
cis-1,2-Dichloroethene	1	50	5	-	1	5	20	50	100	200	300	10
cis-1,3-Dichloropropene	1	50	5	-	1	5	20	50	100	200	300	10
cis-1,4-Dichloro-2-butene	5	250	10	-	1	5	20	50	100	200	300	10
Carbon tetrachloride	1	50	5	-	1	5	20	50	100	200	300	10
Cyclohexane	5	250	5	-	1	5	20	50	100	200	300	10
Chlorobenzene	1	50	5	-	1	5	20	50	100	200	300	10
Chloroethane	1	250	5	-	1	5	20	50	100	200	300	10
Chloroform	5	250	5	-	1	5	20	50	100	200	300	10
Chloromethane	1	50	5	-	1	5	20	50	100	200	300	10
Dibromochloromethane	1	50	5	-	1	5	20	50	100	200	300	10
Dichlorofluoromethane	1	50	5	-	1	5	20	50	100	200	300	10
Diethyl ether (Ethyl ether)	5	50	5	-	1	5	20	50	100	200	300	10
Dichlorodifluoromethane	1	50	5	-	1	5	20	50	100	200	300	10
Diisopropyl ether	1	50	5	-	1	5	20	50	100	200	300	10
Dibromomethane	1	50	5	-	1	5	20	50	100	200	300	10
Ethylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
Ethyl-tert-butyl ether	5	250	5	-	1	5	20	50	100	200	300	10
Hexachloro-1,3-butadiene	5	50	5	-	1	5	20	50	100	200	300	10
Hexachloroethane	5	250	5	-	1	5	20	50	100	200	300	10
Iodomethane	5	250	5	-	1	5	20	50	100	200	300	10
Isopropylbenzene (Cumene)	1	50	5	-	1	5	20	50	100	200	300	10
Isopropyl acetate	5	250	5	-	1	5	20	50	100	200	300	10
Isobutanol	50	2500	50	-	10	50	200	500	1000	2000	3000	100
Methyl acetate	10	250	20	-	1	5	20	50	100	200	300	10
Methylene Chloride	1	50	5	-	1	5	20	50	100	200	300	10
Methylcyclohexane	5	250	5	-	1	5	20	50	100	200	300	10
Methyl-tert-butyl ether	1	50	5	-	1	5	20	50	100	200	300	10
4-Methyl-2-pentanone	5	250	5	-	1	5	20	50	100	200	300	10
m&p-Xylene	2	100	10	-	2	10	40	100	200	400	600	20
Naphthalene	5	250	5	-	1	5	20	50	100	200	300	10
n-Butanol	250	12500	250	-	50	250	1000	2500	5000	10000	15000	500
n-Butylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
n-Heptane	5	250	5	-	1	5	20	50	100	200	300	10
n-Hexane	5	250	5	-	1	5	20	50	100	200	300	10
n-Propylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
n-Propanol	250	12500	250	-	50	250	1000	2500	5000	10000	15000	500
o-Xylene	1	50	5	-	1	5	20	50	100	200	300	10
p-Isopropyltoluene	1	50	5	-	1	5	20	50	100	200	300	10
sec-Butylbenzene	5	50	5	-	1	5	20	50	100	200	300	10
Styrene	1	50	5	-	1	5	20	50	100	200	300	10
trans-1,2-Dichloroethene	1	50	5	-	1	5	20	50	100	200	300	10
trans-1,3-Dichloropropene	1	50	5	-	1	5	20	50	100	200	300	10

Analyte	PQL water µg/L	PQL soil µg/kg	PQL 5035 soil µg/Kg	Cal1 MeOH curve only µg/L	Cal2 MeOH Cal1 water µg/L	Cal3 MeOH Cal2 water µg/L	Cal4 MeOH Cal3 water µg/L	Cal5 MeOH Cal4 water µg/L	Cal6 MeOH Cal5w ater µg/L	Cal7 MeOH Cal6 water µg/L	Cal7 water Curve only µg/L	* Optional Cal3 water Curve only µg/L
trans-1,4-Dichloro-2-butene	5	250	5	-	1	5	20	50	100	200	300	10
tert-Amyl-methyl ether	1	250	5	-	1	5	20	50	100	200	300	10
tert-Butyl Alcohol	25	1250	50	-	5	25	100	250	500	1000	1500	50
Tetrachloroethene	1	50	5	-	1	5	20	50	100	200	300	10
Tetrahydrofuran	5	250	10	-	1	5	20	50	100	200	300	10
Toluene	1	50	5	-	1	5	20	50	100	200	300	10
Trichloroethene	1	50	5	-	1	5	20	50	100	200	300	10
Trichlorofluoromethane	1	50	5	-	1	5	20	50	100	200	300	10
tert-Butylbenzene	1	50	5	-	1	5	20	50	100	200	300	10
Xylene (Total)	3	150	15	-	3	15	60	150	300	600	900	30
Vinyl acetate	5	250	5	-	1	5	20	50	100	200	300	10
Vinyl chloride	1	50	5	-	1	5	20	50	100	200	300	10

*** If the Optional Cal 3 point is made for the Water Curve all subsequent Cal levels will increase by 1 (i.e. 300µg/L is now CAL8)**

11.2.2. Calibration Response Factors

Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard (µg/L).

C_x = Concentration of the compound being measured (µg/L).

Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8260B method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system.

These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples.

11.2.3. Calibration Curve Fit

The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the “goodness of fit”.

Average Response Factor (ARF): The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF (see Table 11.2).

The % RSD is calculated as follows: $\%RSD = SD * 100 / ARF$

Where: SD = Standard deviation of the averaged RFs for a given compound

The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The **Calibration Check Compounds (CCCs)** are a subset of the target analyte list that must meet specific criteria (see Table 11.2) for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria. If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated.

Linear Regression: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y = ax + b$ where “a” is the slope of the line and “b” is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y-axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient “r” is used to determine the acceptability of a linear regressed curve (see Table 11.2)

Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y = ax^2 + bx + c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an

analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

Note: The State of South Carolina does not allow the use of Non-linear regression for compliance samples.

Analytes that have poor purging efficiency or are problematic compounds may require the use of Non-linear regression curves. These may include: Bromomethane, 1-Propanol, Acrolein (2-propenal), n-Butanol, 2-Butanone, Carbon Disulfide, Hexachloroethane, and 1,2-Dibromo-3-chloropropane (DBCP)

Refer to section 11.2.3 for curve fit criteria. Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety and reanalyzed. Re-analysis should be within the same 12-hour time window and must occur within 8 hours of the original analysis.

11.3. Calibration Verification

11.3.1. Second Source Verification (SCV)

In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the “fit” of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{\text{SCV}} - \text{TrueValue}_{\text{SCV}}] / \text{TrueValue}_{\text{SCV}} * 100$$

See Tables 10.6 and 10.7 for details on the preparation of this standard. See Table 11.2 for control criteria.

11.3.2. Continuing Calibration Verification (CCV)

As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked at the beginning of every analytical

sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished by analyzing a midpoint calibration standard (CCV).

The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\% \text{ Difference: } [\text{RF}_{\text{CCV}} - \text{AvgRF}_{\text{CAL}}] / \text{AvgRF}_{\text{CAL}} \times 100$$

$$\% \text{ Drift: } [\text{Result}_{\text{CCV}} - \text{TrueValue}_{\text{CCV}}] / \text{TrueValue}_{\text{CCV}} \times 100$$

Table 11.2 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD \leq 15%	If not met, try linear regression fit
	Linear Regression	$r \geq 0.99$	If not met, try non-linear regression fit
	Non-linear Regression	COD ≥ 0.99	If not met, remake standards and recalibrate
System Performance Check Compounds (SPCCs)	Chloromethane	Avg RF ≥ 0.10	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system.
	1,1-Dichloroethane	Avg RF ≥ 0.10	
	Bromoform	Avg RF ≥ 0.10	
	Chlorobenzene	Avg RF ≥ 0.30	
	1,1,2,2-Tetrachloroethane	Avg RF ≥ 0.30	
Calibration Check Compounds (CCCs)	1,1-Dichloroethene	%RSD $< 30\%$	%RSD for the calibration check compounds (CCC's) must be $\leq 30\%$ regardless of curve fit used. If the CCCs are not included on a list of analytes for a project, and therefore not included in the calibration standards, then all compounds of interest must meet a $\leq 15\%$ RSD criterion.
	Toluene		
	Chloroform		
	Ethylbenzene		
	1,2-Dichloropropane		
Second Source Verification Standard	Immediately after each initial calibration	% Diff $\pm 30\%$	Acceptance criteria are $\pm 30\%$ for all analytes, with allowances for 5% of compounds @ $\pm 40\%$. See ALL_Q_025_Rev.1
	Continuing Calibration Verification		
	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT	RT ± 30 sec	Use midpoint calibration standard as reference
	Internal Standard Response	50 – 200%	Use midpoint calibration standard as reference
	CCCs	RF $\pm 20\%$ Diff. Result $\pm 20\%$ Drift	Use for Avg RF calibration curves Use for linear and non-linear calibration curves Additional client specific requirements for the analysis of contract samples requires that BTEX, PAH, Oxygenates, and surrogate compounds also be considered CCCs and must meet the 20% CCV criterion.
	Non-CCC Targets	RF $\pm 50\%$ Diff.* Result $\pm 50\%$ Drift	Some programs may require control over non-CCC target analytes. In the absence of specified criteria, use those listed *State of South Carolina requires non-CCC Compounds to meet $\pm 30\%$ Drift. Please Note: Analytes that have poor purging efficiency or are problematic compounds may require the use $\pm 50\%$ Drift. These may include: Bromomethane, 1-Propanol, Acrolein (2-propenal), n-Butanol, 2-Butanone, Carbon Disulfide, Hexachloroethane, and 1,2-Dibromo-3-chloropropane (DBCP)

11.4. Calibration Corrective Actions

11.4.1. Calibration Linearity Problems

- 11.4.1.1. Check instrumentation/equipment condition.
- 11.4.1.2. Enter maintenance in instrument maintenance logbook.
- 11.4.1.3. Perform another initial calibration.
- 11.4.1.4. No data can be reported.
- 11.4.1.5. Generate on Non-Conformance Memo.

11.4.2. Secondary Verification Problems

- 11.4.2.1. Check instrumentation/equipment condition.
- 11.4.2.2. Enter maintenance in instrument maintenance logbook.
- 11.4.2.3. Perform another initial calibration.
- 11.4.2.4. No data can be reported.
- 11.4.2.5. Generate on Non-Conformance Memo

11.4.3. Continuing Verification Problems

- 11.4.3.1. Reanalyze the original CCV standard to determine instrument consistency.
- 11.4.3.2. Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.
- 11.4.3.3. Document instrument maintenance
- 11.4.3.4. Reanalyze CCV standard to determine if maintenance was effective in restoring performance.
- 11.4.3.5. Complete recalibration of instrument.
- 11.4.3.6. If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the injection log with a thorough explanation for the deviation from policy.

Exceptions: If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

12. Procedure

12.1. Purge-Trap GC/MS System Preparation

12.1.1. Operating Parameters

Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 12.1 –Instruments and Operating Parameters

Component	Settings and Consumables	
Gas Chromatograph	Column: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 µm Inlet Liner: Restek 4 mm Single Gooseneck Injection Port Liners Inlet Seal: Restek Gold Plated inlet seal Column Ferrules: Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5-1.0 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
Mass Spectrometer	Tune File: Named to date of tune	
Purge & Trap Concentrator	Prepurge: NO Preheat: 40°C Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 5-7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
Autosampler	Syringe Flushes: 2 Spurge Tube Flushes: 2	

12.2. Tune Verification

At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing bromofluorobenzene (refer to table 10.2). The tune verification standard can be combined with the CCV standard provided that the amount of BFB introduced into the system meets the criteria in Section 12.2.

After the analysis of this standard, the mass spectrum of BFB must be evaluated against the following criteria.

Mass (m/z)	Ion Abundance criteria
50	15.00-40.00% of m/z 95
75	30.00-60.00% of m/z 95
95	Base peak, 100% relative abundance
96	5.00-9.00% of m/z 95
173	<2.00% of m/z 174
174	50.00-100.00% of m/z 95
175	5.00-9.00% of m/z 174
176	95.00-101.00% of m/z 174
177	5.00-9.00% of m/z 176

To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for bromofluorobenzene. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Additionally, see Attachment III and Attachment IV on the proper techniques for evaluation of the tune file. Otherwise, the spectra must be obtained in one the following manners, in the listed order.

1. Using an average of three scans, centered on the apex of the peak; or,
2. Using an average of all scans across the width of the peak, taken at half height; or,
3. Using an average of all scans taken across the width of the peak from baseline to baseline.

A background scan taken immediately before but not including the peak must be subtracted.

Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the BFB tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the BFB tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

If the ratios do not meet the criteria, refer to the following corrective actions to address the problem:

1. Retune the mass spectrometer following the equipment manufacturers' instructions. The tune status must be verified after the tuning procedures.
2. If this fails, change filament and retune.
3. If this fails, take down the mass spectrometer and clean the instrument.

12.3. Calibration Verification

After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 11.2 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 11.4.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

Note: In situations where the instrument will run unattended (i.e. overnight), the analyst may load sequential CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, the CCV must be evaluated according to the protocol set forth in the Quality Assurance Manual within Section 6 – Equipment and Measurement Traceability.

12.4. Operation of the Software Systems

12.4.1. Epic Pro

12.4.1.1. Make Q-Batch

- 12.4.1.1.1. Batching -> New Batch -> Queue = MSV
- 12.4.1.1.2. Click Empty Batch icon on taskbar
- 12.4.1.1.3. Highlight QC Rule -> F9 -> type MSV
- 12.4.1.1.4. Select appropriate QC Rule (i.e. MSV water) – Select OK – F10 to save
- 12.4.1.1.5. Record Q Batch #

12.4.1.2. Create Standards

- 12.4.1.2.1. System -> Utility -> Clone Standard by Event
- 12.4.1.2.2. Select Event (111 = MeOH soil curve, 115 = Water/LLsoil curve)
Select OK
- 12.4.1.2.3. Double Click on standard event
- 12.4.1.2.4. Review Standard composed of – Find/Replace if necessary
- 12.4.1.2.5. Update expiration date to 7 days from creation – F10 to Save
- 12.4.1.2.6. Operations -> Standard Log -> Enter – Record Standard #'s

12.4.2. Chemstation

12.4.2.1. Create Chemstation methods

- 12.4.2.1.1. Tune MS, Save Tune file as date (i.e. 072513.u)
- 12.4.2.1.2. Update both DBFB and Curve method to use new tune file
- 12.4.2.1.3. Save Curve method as date (i.e. W072513.m)

12.4.2.2. Set up Sequence

- 12.4.2.2.1. Load pre-existing curve sequence if available
- 12.4.2.2.2. Change old method to the new method & copy through all files

(DBFB remains the same)

12.4.2.2.3. Change Q-Batch # in BFB, Curve and ICV files

12.4.2.3. **Start Analysis**

12.4.2.3.1. Run minimum of 2 BFB injections to ensure the tune is optimized

12.4.2.3.2. Retune or adjust as needed, repeat 2 more BFB

12.4.2.3.3. Analyze a 2 blanks to verify the system is clean and IS areas within range

12.4.2.3.4. First IS, pentafluorobenzene should be between 300,000 – 550,000 area counts

12.4.2.3.5. Raise or lower EM as necessary – You **Must** reanalyze BFB if voltage was adjusted

12.4.2.3.6. Reanalyze blanks to ensure correct voltage and proceed w/ analysis of curve

12.4.3. **Target**

12.4.3.1. **Create Method**

12.4.3.1.1. Rename existing method to new name matching Chemstation method (i.e. W072513.m)

Note: if other data in Directory was processed w/ old method a copy of that method must remain in directory as well.

12.4.3.1.2. To avoid excessive file size, Audit trail in method should be reset at a minimum of annually

The **ONLY** time an audit trail may be reset is prior to calibrating the instrument.

Note: The Audit trail will remain intact in previous day's folder.

Double click into method folder, highlight the .audit file and delete

12.4.3.2. **Edit Method**

12.4.3.2.1. Security -> Method unlocked

12.4.3.2.2. Global -> Calibration – Click “update Curve Parameters” to averaged

12.4.3.2.3. File -> Zero Calibration

12.4.3.2.4. Compound -> Edit Compound -> Calibration

12.4.3.2.4.1. Review all analytes to ensure all necessary points are enabled

12.4.3.2.4.2. Are any 300 points dropped? If so, mark them enabled and make note of these to change the “Max Compound Amount Limit” after the curve has been run.

12.4.3.2.5. Reports -> Tabular -> “Print Custom Report” – click “Select Format”

12.4.3.2.5.1. On toolbar a “Select” icon will appear

12.4.3.2.5.2. Click on ManIntprepostRev.mac – click “Open”

Note: It is necessary to do this **Every** time a calibration is zeroed, even if the macro shows up in this field as the link to the macro that was lost when the calibration was zeroed.

12.4.3.2.6. Sample -> Default Sample

12.4.3.2.6.1. Change “Lab Prep Batch” field to the new Q-Batch #

12.4.3.2.6.2. Change “Client SDG” to be the instrument and date (i.e. 40MSV2-07252013)

12.4.3.2.7. Sample -> Surrogate/ISTD Parameter

12.4.3.2.7.1. Confirm that the correct IS/SS standard # is entered in the “Surrogate Lot#” field

- 12.4.3.2.7.2. Example - 51970:1.163 The 51970 is the IS/SS number followed by a colon followed by the volume added (this is a fixed amount unless change to the standard delivery has occurred.)
- 12.4.3.2.8. File -> Save Method
- 12.4.3.2.9. File -> Exit
- 12.4.3.3. **Process and Review Curve Data**
 - 12.4.3.3.1. If significant Column maintenance was performed, it may be beneficial to process the 20 or 50 point first to update RT's as the larger concentrations will have better spectra to confirm correct identification
 - 12.4.3.3.2. Select Method to calibrate and process files
 - 12.4.3.3.2.1. Compound Sublist should be "all.sub"
 - 12.4.3.3.2.2. Sample Type change to Calib Sample
 - 12.4.3.3.2.3. Cal Level change to appropriate level 1-7
 - 12.4.3.3.2.4. Double check that the Q-Batch # in MiscInfo and Lab Prep Batch are correct and match
 - 12.4.3.3.2.5. Double check that the Client SDG reflects the instrument and date
 - 12.4.3.3.3. Review Target Data
 - 12.4.3.3.3.1. Review each analyte of all points for correct spectrum, RT and appropriate integration
 - 12.4.3.3.3.2. All Manual Integration of all curve points and ICV need to have Review Codes added
 - 12.4.3.3.3.3. After reviewing all points, review each analyte point 1 -> 7 to ensure consistent RT, spectra and Integration (i.e. shoulders cropped or included, etc.)
- 12.4.3.4. **Review Curve in Target Method**
 - 12.4.3.4.1. Edit Method
 - 12.4.3.4.2. Edit Compound -> Calibration
 - 12.4.3.4.3. Review each analyte to ensure Initial Calibration % RSD are less than 15.0%
 - 12.4.3.4.3.1. Note analytes > 15% and re-examine target data for proper integration
 - 12.4.3.4.4. Check that all CCC compounds are less than 30% RSD
 - 12.4.3.4.4.1. CCC's are 11DCE, chloroform, 12DiChloropropane, toluene, ethylbenzene and vinyl chloride
 - 12.4.3.4.4.2. Instrument maintenance must be performed to correct problem if any >30%
 - 12.4.3.4.4.3. If RSD >15% and <30% note %RSD to record later
 - 12.4.3.4.5. Check that all minimum relative response factors (RRF) were met for the SPCC – Chloromethane, 11DCE, bromoform are 0.1 and 1122PCA, chlorobenzene are 0.3 – if any %RSD >15 not RRF to record later
 - 12.4.3.4.6. If %RSD >15% - Drop Upper or lower point to achieve %RSD <15
 - 12.4.3.4.6.1. If the Report Limit (RL) for analyte is not the 1 point, can the 1 point be disabled
 - 12.4.3.4.6.2. Can the 7 point be dropped (or 6&7 points) - ** Will require lowering Max Amount
Note: ONLY upper or lower points can be dropped, *Never* an intermediate point!!
 - 12.4.3.4.6.3. Must have minimum of 5 points for Averaged RF curve
 - 12.4.3.4.6.4. After disabling appropriate points – Click "Update Calibration" button

- 12.4.3.4.7. Is %RSD still >15 – Switch Curve fit to Linear Regression
 - 12.4.3.4.7.1. Change curve fit to Linear
 - 12.4.3.4.7.2. **CCC Compounds (11DCE, chloroform, 12dichloropropane, toluene, ethylbenzene, vinyl chloride) MUST still be <30% RSD.
 - 12.4.3.4.7.3. Initial Calibration R^2 must be 0.990 or greater
 - 12.4.3.4.7.4. Must have minimum of 5 points for Linear regression curve
 - 12.4.3.4.7.5. b intercept should be as close to zero as possible
 - 12.4.3.4.7.5.1. i.e. by dropping the 300 point does the intercept go from 0.1980442 -> 0.0681234
 - 12.4.3.4.7.5.2. This will give less false positive hits but require linear range to be lowered to 2000 µg/L
- 12.4.3.4.8. If R^2 is not >0.990
 - 12.4.3.4.8.1. Change curve fit to Quadratic
 - 12.4.3.4.8.2. ***Must have minimum of 6 points**
 - 12.4.3.4.8.3. R^2 must be 0.990 or greater
 - 12.4.3.4.8.4. Like Linear regression the 300 point can be dropped (or 1 point added if RL is 5 µg/L) to achieve the intercept closest to zero, as long as 6 points remain and linear range is adjusted.
- 12.4.3.4.9. If calibration for compound will not pass
 - 12.4.3.4.9.1. The instrument cannot be run for lists including these analytes
 - 12.4.3.4.9.2. Document analytes as failing in Run logbook
 - 12.4.3.4.9.3. Place Post-It-Note on Instrument Terminal to alert other analysts of failures
- 12.4.3.5. **Update Linear Range**
 - 12.4.3.5.1. After all analyte curve fits have been checked
 - 12.4.3.5.1.1. Compound ->Edit Compound -> Report Parm
 - 12.4.3.5.1.2. Adjust “Max Compound Amt Limits” to reflect highest point used (300->200 if 7th point was dropped)
 - 12.4.3.5.2. Sublists -> Update Sublists
 - 12.4.3.5.2.1. Check the “Update Sublists QC Limits” box
 - 12.4.3.5.2.2. Highlight first sublist and hit Enter button
 - 12.4.3.5.2.3. Arrow down to the next sublist and hit Enter
 - 12.4.3.5.2.4. Repeat for all Sublists
 - 12.4.3.5.2.5. **If you fail to update all the sublists, detects above linear range will not be “a” flagged in target.
 - 12.4.3.5.2.5.1. EpicPro used the “a” flag to switch Condition Code from “OK” to “OR”
- 12.4.3.6. **Lock Method**
 - 12.4.3.6.1. Security -> Initial Calibration Locked
 - 12.4.3.6.2. Note: Do not select “Method Locked” – This would not allow the method to be used to process data
- 12.4.3.7. **Verify Initial Calibration**
 - 12.4.3.7.1. View -> Initial Calibration
 - 12.4.3.7.2. This generates a report with calibration data that will appear on the lower tool bar
 - 12.4.3.7.3. Print report and review
 - 12.4.3.7.3.1. The Calibration File Names in the header match the **correct** files used in the curve
 - 12.4.3.7.3.2. All Average Response Factors < 15% and at least 5 points were included
 - 12.4.3.7.3.3. All Linear Regression >0.990 and at least 5 points were included

- 12.4.3.7.3.4. All Quadratic > 0.990 and at least 6 points were included
- 12.4.3.7.3.5. Are all low points dropped below Report Limit for that analyte
- 12.4.3.7.3.6. Any high points dropped verify that the Max on Column was lowered and Record max amount on the report
- 12.4.3.7.3.7. No midpoints of curve are missing
- 12.4.3.7.3.8. All CCC compounds averaged – If not is the %RSD < 30% - Record actual RSD on report
- 12.4.3.7.3.9. All SPCC minimum RF factors met – If not averaged, switch to Averaged in method record the RF on the report and switch curve back to appropriate curve fit
- 12.4.3.7.4. Manually check individual Response Factors (RF) for at least one analyte
 - 12.4.3.7.4.1. Calculation the RF for each point in the curve of an Averaged curve fit using the following formula
 - 12.4.3.7.4.1.1. $RF = (\text{Area of analyte} \times \text{concentration of IS}) / (\text{Area of IS} \times \text{concentration of analyte})$
- 12.4.3.7.5. Save method and Exit
- 12.4.3.8. **Re-quantify and Uploading Curve and ICV**
 - 12.4.3.8.1. Select Method
 - 12.4.3.8.2. Highlight Curve and re-quantitate
 - 12.4.3.8.3. Process ICV – Must use all.sub (or Full.sub)
 - 12.4.3.8.4. Review ICV and check CLP.rp
 - 12.4.3.8.4.1. All SPCC Minimum RF must be met (if analyte is linear, must hand calculate)
 - 12.4.3.8.4.2. All CCC Analytes must be <20%
 - 12.4.3.8.4.3. All other analytes must be <30%
 - Note: Up to 5% (5 Analytes for a full list spike) may be between 30-40%)
 - 12.4.3.8.4.4. All Analytes >40% will be flagged as failing
 - 12.4.3.8.4.4.1. Document analytes as failing in Run logbook
 - 12.4.3.8.4.4.2. Place Post-It-Note on Instrument Terminal to alert other analysts of failures
 - 12.4.3.8.5. Generate all files to paperless (BFB, Curve and ICV)
 - 12.4.3.8.6. Upload all files to EpicPro (double check Q-Batch is correct prior to upload)
 - 12.4.3.8.7. Check Q-Batch in Epic to ensure Curve, BFB, and ICV imported correctly (may take several minutes)
- 12.4.3.9. **MN Low Standard Verification**
 - 12.4.3.9.1. Copy 1ppb, 5ppb, & 20ppb files into another folder (i.e. the unprocessed blank following 300ppb)
 - 12.4.3.9.2. Paste all 3 files than Rename (example 07251305.D -> MN01-07251305.D)
 - 12.4.3.9.2.1. This will allow original files to be un-manipulated
 - 12.4.3.9.3. Cut files and paste back in original folder
 - 12.4.3.9.4. Re-Quant new MN files as LCS
 - 12.4.3.9.4.1. Sample Type = QC Control Sample
 - 12.4.3.9.4.2. Click QC Sample Type
 - 12.4.3.9.4.2.1. Sample Type = LCS
 - 12.4.3.9.4.2.2. Spike List = MNLOW1.spk, MNLOW5.spk, MNLOW20.spk
 - 12.4.3.9.5. Highlight all 3 files and Do Quick Forms – Form 3 of LCS

12.4.3.9.6. Print Form 3.s and pass on to Supervisor to update MN report limits in EpicPro

12.4.3.10. **Before proceeding with analysis of samples**

12.4.3.10.1. Check Chemstation sequence that correct Q-Batch is in BFB and CCC

12.4.3.10.2. Check that correct Method is referenced in the sequence

12.5. Sample Preparation

12.5.1. Samples

12.5.1.1. Sample Pre-screening

12.5.1.1.1. Samples are pre-screened using a rapid GC headspace technique. See SOP S-GB-O-001 *Sample Screening Volatile Organics Prior to Preparation* (most current revision or replacement) for the specifics on the pre-screening of samples.

12.5.1.2. Water Samples

After pre-screening, water samples typically do not require any sample preparation unless they require a dilution to bring high-level contaminants within calibration range or to minimize matrix interference. Dilutions are made following Section 12.5.1.5.1.

After analysis check the residue in the vial following analysis using pH paper. The pH should be <2. Document results in the run sequence log as <2 or >2. Footnote any sample not meeting the pH requirement. If dilutions are required, pH preservation can be verified at the time the dilution is made using the sample remaining in the original sample container.

12.5.1.3. Soil Samples

12.5.1.3.1. Low concentration soils

Samples received for low level analysis should be contained in pre-weighed VOA vials either with or without Reverse Osmosis Water (ROW) and/or sodium bisulfate preservative. NOTE: some samples may be received in coring devices (e.g. Encore™, etc.). These samples must be extruded into a VOA vial either with or without ROW and/or sodium bisulfate and a magnetic stir bar within 48 hours of sample collection. If samples are received that are greater than 10g the PM must be notified and samples will be rejected for analysis.

12.5.1.3.1.1. **Weight determination:** Prior to preparation or analysis of any soil received in a pre-weighed VOA vial, the sample weight must be determined and recorded. Accurately weigh the VOA vial to 0.01 g in the laboratory; record this amount in the sample preparation logbook. Subtract the tare weight recorded on the vial and 0.18g for each Pace Sample label that was affixed to the pre-tared vial; this will be the weight of sample in the vial.

12.5.1.3.1.2. Samples received pre-weighed in the field must be in 40 mL VOA vials and contain a magnetic stir bar, acid preservative and a

field tare weight. The analyst will compare the field tare weight to the weight of the sample before analysis. The weight of the sample should be recorded.

12.5.1.3.1.3. All samples must be extruded from the coring devices within 48 hrs. of collection. If the samples are to also be analyzed within the 48 hr criteria, no acid preservation is required. If analysis is to occur after 48 hrs. but within 14 days, the samples must be preserved with Sodium Bisulfate or if preserved with ROW, stored frozen. The ratio of Sodium Bisulfate to sample weight is 0.2g of preservative to 1g of sample.

12.5.1.3.2. **High concentration soils**

12.5.1.3.2.1. **Methanol-Preserved Samples:** Samples received in pre-weighed vials preserved with methanol must be accurately weighed in the laboratory to 0.01 g and the sample weight determined. See Section 12.5.1.4.2.1.2. Subtract the tare weight written on the VOA vial and 0.18g for each Pace Sample label that was affixed to the pre-tared vial from the weight determined in the laboratory. This will be the weight of sample in the VOA vial. The volume of methanol in the sample container should be at a 1 to 1 ratio of soil to methanol.

12.5.1.3.2.1.1. **Calculation of 1:1 ratio soil (g) to MeOH (mL).**

To calculate the amount of soil weight (g) in the sample can be calculated as follows:

Where:

$$W_s = W_t - W_j - (N * W_l)$$

W_s = Weight of soil in the sample (g)

W_t = Total weight of the sample including vial, cap, soil, and MeOH (g)

W_j = Weight of the jar including the vial, cap, and MeOH (g)

N = Number of Pace Sample labels

W_l = Weight of Pace Sample label affixed to the pre-tared 40mL VOA vial; which has been determined to be 0.18g

To calculate volume of MeOH to achieve the 1:1 ratio of soil weight (g) to MeOH (mL) may be calculated as follows:

Where:

$$V_M = W_s - 10\text{mL}$$

V_M = Volume of MeOH required to achieve 1:1 ratio of soil (g):MeOH (mL)

W_s = Weight of soil in the sample (g)

10 = Volume of MeOH initially added (mL)

12.5.1.3.2.2. **Unpreserved Samples:** Samples received in unpreserved pre-weighed vials must be accurately weighed in the laboratory to 0.01 g. NOTE: some samples may be collected and transported to the laboratory in bulk containers. An accurately weighed ≥ 5 gram sub-sample must be taken and added to a 40mL VOA vial. The non-compliant sample collection technique must be recorded in the preparation logbook and a qualifier added to the sample result. The samples are then preserved with 10 mL methanol within 48 hours of sample collection. To determine the sample weight: subtract the weight written on the VOA vial from the weight determined in the laboratory prior to the addition of the 10 mL methanol preservative. This will be the weight of sample in the VOA vial.

12.5.1.3.2.3. The balance is to be leveled before calibration. Calibration verification of the analytical balance is done with S-class weights. These values are to be noted in the Balance calibration logbook. The frequency of balance calibration verification is once per day before the balance is used or when the balance is moved.

12.5.1.4. **Dilutions**

12.5.1.4.1. **Water**

Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots may be measured in either a volumetric pipette or syringe and brought to volume in a volumetric flask.

12.5.1.4.1.1. All steps must be performed without delays until the diluted sample is in a 40 mL VOA Vial.

12.5.1.4.1.2. Dilutions are made in gastight 50mL syringes.

12.5.1.4.1.3. Calculate the approximate volume of organic-free reverse osmosis water (ROW) added to the syringe and add slightly less than this quantity of ROW to the syringe barrel.

12.5.1.4.1.4. Inject the proper aliquot of sample using the appropriate 10 μ L to 5mL syringes to create the desired dilution in the 50 mL syringe. Dilute the sample to the mark with ROW. Invert, and rock back and forth three times. Repeat the above procedure for additional dilutions.

12.5.1.4.1.5. Fill a 40mL VOA vial with the diluted sample from the 50 mL syringe prepared in Section 12.5.1.5.1.4.

12.5.1.4.1.6. Place the VOA vial on the autosampler. All dilutions should keep the response of a major constituent (previously saturated peaks) in the upper half of the linear range of the curve.

12.5.1.4.1.7. The autosampler will add the internal standard and surrogate to the sample and transfer 5 mL over to the 5 mL sparge tube on the concentrator.

12.5.1.4.2. **Soil**

12.5.1.4.2.1. **Low Level Soils**

12.5.1.4.2.1.1. It will be necessary to adjust the sample weight for

quantitation purposes. Any analyte hits outside of the calibration range, 200 µg/kg, will be extracted into Methanol and analyzed under High Concentration Sample criteria.

12.5.1.4.2.2. **High Concentration Soils**

12.5.1.4.2.2.1. Dilute all samples according to the results of the screening data. A standard analytical dilution is 1:50. Add 1.0 mL of the sample extract measured with a microsyringe of appropriate volume to 49 mL of reverse osmosis water in a 50 mL syringe.

12.5.1.4.2.2.2. To make dilutions other than a standard 1:50 dilution, fill a 50 mL syringe to a volume of 49 mL with reverse osmosis water. Using a 1.0 mL syringe, inject methanol into the 50 mL syringe to bring the total volume of sample and methanol to equal 1.0 mL. Using an appropriate volume syringe, inject the sufficient amount of sample to reach desired dilution. Invert, and rock back and forth three times.

12.5.1.4.2.2.3. The 50 mL syringe contents are placed into a 40 mL VOC vial by slowly deploying the plunger and injecting on the side of the vial to eliminate cavitation and loss of analytes to volatilization or sparging. Enough of the contents are injected to create a meniscus at the top of the vial that when capped will produce a no headspace sample.

12.5.1.4.2.2.4. The vial is capped and checked for headspace. If vial is free of headspace, it is ready for analysis as per Section 14.

12.5.2. **Batch QC**

Refer to Table 13.1 for details on Batch QC requirements.

12.5.2.1. **Method Blank**

12.5.2.1.1. **Water**

12.5.2.1.1.1. Fill a 40mL VOA vial with reverse osmosis water (ROW) and place in autosampler rack. The autosampler will add the internal standard and surrogate to the sample and transfer 5mL over to the 5mL sparge tube on the concentrator.

12.5.2.1.1.2. When leach samples are present, one leach blank must be analyzed with the analytical batch in addition to the method blank.

12.5.2.1.2. **Low Level Soil**

12.5.2.1.2.1. A method blank is prepared with 5 mL of ROW into a 40 mL VOA vial containing a disposable magnetic stir bar. The vial is placed onto the autosampler and the autosampler will add the internal standard and surrogate to the sample. The blank is preheated to 40°C and purged. The method blank must be analyzed under the same criteria as the samples.

12.5.2.1.3. **High Concentration Soil**

12.5.2.1.3.1. The method blank (extraction blank) is made by adding 10 µL of

the 2500 ppm surrogate standard in 10 mL methanol placed in a 40 mL VOA vial containing 10g of Ottawa sand. A 1.0 mL portion of this is diluted with 49 mL of ROW for a final concentration on the instrument of 50 µg/L.

12.5.2.2. Laboratory Control Sample/Laboratory Control Sample Duplicate

- 12.5.2.2.1. See Table 10.7 for spiking procedures and Table 13.1 for Batch Quality Control Criteria.
- 12.5.2.2.2. A Laboratory Control Sample Duplicate is required when sample volume for the Matrix Spike/Matrix Spike Duplicate is not received.
- 12.5.2.2.3. When EPA 624 samples are present with SW846 8260B samples, one LCS/D at 20 µg/L and one LCS/D at 50 µg/L must be analyzed to meet each method requirement. If the 20µg/L LCS/D is valid, the additional 50 µg/L LCS/D pair is not required.

12.5.2.3. MS/MSD Samples

- 12.5.2.3.1. See Table 10.7 for spiking procedures and Table 13.1 for Batch Quality Control Criteria..
- 12.5.2.3.2. When one to 20 EPA 624 samples are present one MS/MSD pair should be analyzed for every 20 samples (or one spiked sample per month) at a concentration of 20µg/L.
- 12.5.2.3.3. When Leach samples are analyzed, one MS must be analyzed per each sample matrix submitted for leaching.

13. Quality Control

13.1. Instrument Quality Control: Refer to Table 11.2 for initial and continuing calibration criteria and corrective actions.

13.2. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reverse Osmosis water (ROW)	One (1) per 20 samples or 12 hour window (whichever is most frequent)	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	Re-analyze associated samples. Exceptions: 1. If sample ND, report sample without qualification 2. If sample result >20x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination. 3. If sample result <20x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Method specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene <u>OR (alternative)</u> Full Target List compounds	One (1) per batch of up to 20 samples	Laboratory derived limits <u>Method Specified List:</u> All compounds must pass control criteria, with no exceptions. <u>Full Target List:</u> Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	Analyze a new LCS If problem persists, check spike solution Perform system maintenance prior to new LCS run Exceptions: 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. Note: The State of South Carolina does not allow the use of Marginal Exceedances.
Laboratory Control Sample Duplicate (LCSD)	Method specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene <u>OR (alternative)</u> Full Target List compounds	One (1) per batch of up to 20 samples	Laboratory derived limits <u>Method Specified List:</u> All compounds must pass control criteria, with no exceptions. <u>Full Target List:</u> Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	Analyze a new LCSD If problem persists, check spike solution Perform system maintenance prior to new LCSD run Exceptions: 1) If LCSD rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers. Note: The State of South Carolina does not allow the use of Marginal Exceedances.
Matrix Spike (MS)	Method specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene <u>OR (alternative)</u> Full Target List compounds	One (1) per batch of up to 20 samples, must include one TCLP MS for any analyzed in sequence	Laboratory derived limits	If LCS/LCSD and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate <u>OR (alternative)</u> Sample Dup	One (1) for every 5% of all environmental samples	Laboratory Derived Limits	Report results with an appropriate footnote.

13.3. Sample Quality Control

Table 13.2 – Sample Quality Control criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	Pentafluorobenzene 1,4 Difluorobenzene 1,4-Dichlorobenzene-d4 Chlorobenzene-d5	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	Retention Time: RT must be ± 30 seconds from last calibration check on all samples	Retention Time Failure: 1. If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting. 2. Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	Dibromofluoromethane Toluene-d8 4-Bromofluorobenzene	Added to all samples, spikes, control samples and method blanks prior to analysis	Laboratory derived limits	1. Check system parameters 2. Identify and correct likely cause 3. Re-run samples <u>Exceptions:</u> 1. Surr rec above criteria and target compounds < RL, result may be reported with appropriate footnote. 2. Surr rec out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote.

14. Data Analysis and Calculations

14.1. Analyze Samples

14.1.1. Water Samples

14.1.1.1. Create run sequence log. Place 40 mL VOA vial containing sample (Section 12.5.1.2), or appropriately diluted 40 mL VOA vial containing sample (Section 12.5.1.5.1) onto the autosampler. The autosampler will add the internal standard and surrogate to the sample and transfer 5 mL over to the 5 mL sparge tube on the concentrator.

14.1.2. Low-level Samples

14.1.2.1. Create run sequence log. Place the already weighed 40 mL VOA vial containing the sample, stir bar and 5 mL of reverse osmosis water (ROW) or Sodium Bisulfate solution (Section 12.5.1.4.1) onto the autosampler where another 5 mL of ROW and internal standard and surrogate will be added by the autosampler. The sample is preheated to 40°C and purged. The stir bar is moving continuously during the purge cycle. This also helps in compound recovery by breaking down any clumps that may remain in the sample.

It will be necessary to adjust the sample weight for quantitation purposes. Any analyte hits outside of the calibration range, 200 µg/kg, will be extracted into Methanol and analyzed under High Concentration Sample criteria.

14.1.3. High Concentration Samples

14.1.3.1. Create run sequence log. Place 40 mL VOA vial containing sample (Section 12.5.1.4.2), or appropriately diluted 40 mL VOA vial containing sample (Section 12.5.1.5.2.2) onto the autosampler. The autosampler will add the internal standard to the sample and transfer 5 mL over to the 5 mL sparge tube on the concentrator

14.2. Data Reduction

14.2.1. Qualitative Analysis

Retention Time Comparison: The relative retention time (RRT) of the sample component must be within ± 0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co eluting components.

Mass Spectrum Comparison: The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.
- Compare computer-matched compounds with reference spectra to accept or reject each identification.
- All ions present in the reference spectrum that are at least 10% of the base peak must be present in the sample background-subtracted spectrum.
- The relative intensities of these ions must agree within $\pm 30\%$ between the standard and sample spectra.
- While this is a good guideline, acceptance or rejection will depend upon the judgment of the analyst\

Carry-Over Protocol: Each sample must be closely evaluated to confirm that reported values are not a result of carry-over from a previous sample or QC standard. The blank(s) following the CCV or batch QC can be evaluated to determine typical amounts of carry-over to be expected from a sample with results around the mid-range of the curve (i.e. naphthalene and hexachlorobutadiene may carry over 0.5-2ppb from a 50ppb detect whereas vinyl chloride or ketones generally do not have any carry-over at the same concentration). Additionally, the blank(s) following the upper point of an ICAL can demonstrate carry-over of compounds at the upper end of the curve. Obviously, carry-over results vary from instrument to instrument and even ICAL to ICAL; therefore, when there is any question of carry over, an example of similar hits on that particular instrument's recent analysis are to be considered (i.e. a sample earlier in the sequence had benzene hit of 200ppb and the following sample was ND; therefore, it would be reasonable to assume

that a benzene detect of 1.5ppb following a sample containing 100ppb of benzene was not a result of carry-over). Every possible attempt to avoid carry-over contamination in clients' samples will be taken. This may include running instrument clean up blanks following standards that are known to carry-over or following samples that are known to have high concentrations of contaminants, or attempting to run groups of similarly concentrated samples together instead of intermittently through a sequence. When highly contaminated samples are grouped together it is to be expected that a percentage of a reported value may result from carry-over, in this case the significance of carry-over compared to reported value must be considered (i.e. a hit of PCE typically may carry-over 0.5ppb from a sample with an on-column concentration of 100ppb; therefore, a sample with concentration of 70ppb following a sample with a concentration 100ppb may contain roughly 0.5ppb of PCE resulting from carry-over which should be deemed insignificant). Similar to rules concerning Method Blank contamination, if the reported result is greater than 20 times the expected carry-over concentration, the resulting carry-over should be considered insignificant and value acceptable to report.

14.3. Quantitative Analysis – Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

Raw Data Results: The GC/MS data system will calculate the concentration of each analyte as µg/L (or ng/mL). For water samples, no further calculations are necessary unless a dilution of the sample has been performed. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

14.4. Calculation – Aqueous Sample:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

Where:

A_x = Area of characteristic ion for compound being measured.

I_s = Amount of internal standard injected (ng).

A_{is} = Area of characteristic ion for the internal standard.

RF = Average Relative Response factor for compound being measured.

V_o = Volume of water purged (mL), taking into consideration any dilutions made.

14.5. Soil/ Solid calculations:

$$\text{High Conc. (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)}$$

$$\text{Low Conc. (ug/kg)} = \frac{(A_x)(I_s)}{(A_{is})(RF)(W_s)}$$

Where:

A_x , I_s , A_{is} , RF = Same as in water and water-miscible waste above.

V_t = Volume of total extract (mL).

V_i = Volume of extract added (mL) for purging.

S_v = Volume of diluted extract.

W_s = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

14.6. Tentatively Identified Compounds (TICs) – For some samples, identification may be desired for non-target compounds. A mass spectral library search may be conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within $\pm 20\%$.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in Section 13.

16. Corrective Actions for Out-of-Control Data

16.1. See table in Section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the table in Section 13, the contingencies are as follows. If there is no additional volume to perform analyses, all data will be reported as final with applicable qualifiers.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement) for each matrix per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement).

18.2.1. Analysis of four (4) LCS for each matrix the analyst will be performing. The concentration for low level soil and methanol soils should be at the current LCS spike concentration and the recovery is to be within the current LCS QC limits. The concentration for aqueous DOC should be at 20 µg/L and the recovery is to be within the current LCS QC limits.

19. Method Modifications

Method modifications for EPA method 8260B and EPA 624 are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- If a client fails to provide the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the “M5” data qualifier.

Method modifications for EPA method 5035 is as follows:

- The laboratory uses a modification of SW-846 Method 5035 for medium-level volatiles in soil. The laboratory uses 10 grams of soil and 10 mL of methanol whereas the method indicates 5 grams of soil and 5 mL of methanol.

- *Note:* Samples reported to the State of South Carolina requires the use of 5 grams of soil and 5 mL of methanol.

20. Instrument/Equipment Maintenance

20.1. See current version of SOP: S-GB-Q-008 *Preventative, Routine, and Non-routine Maintenance*.

21. Troubleshooting

21.1. See most current version of the Instrument Operations Manual.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. Equipment: Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

- 25.1. Pace Quality Assurance Manual- most current version.
- 25.2. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.
- 25.3. USEPA, SW-846, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.
- 25.4. USEPA, SW-846, Method 5030B, "Purge and Trap for Aqueous Samples," December 1996.
- 25.5. USEPA, SW-846, Method 5035A, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1 July 2002.
- 25.6. USEPA, SW-846, Method 8000B, "Determinative Chromatographic Separations", December 1996.

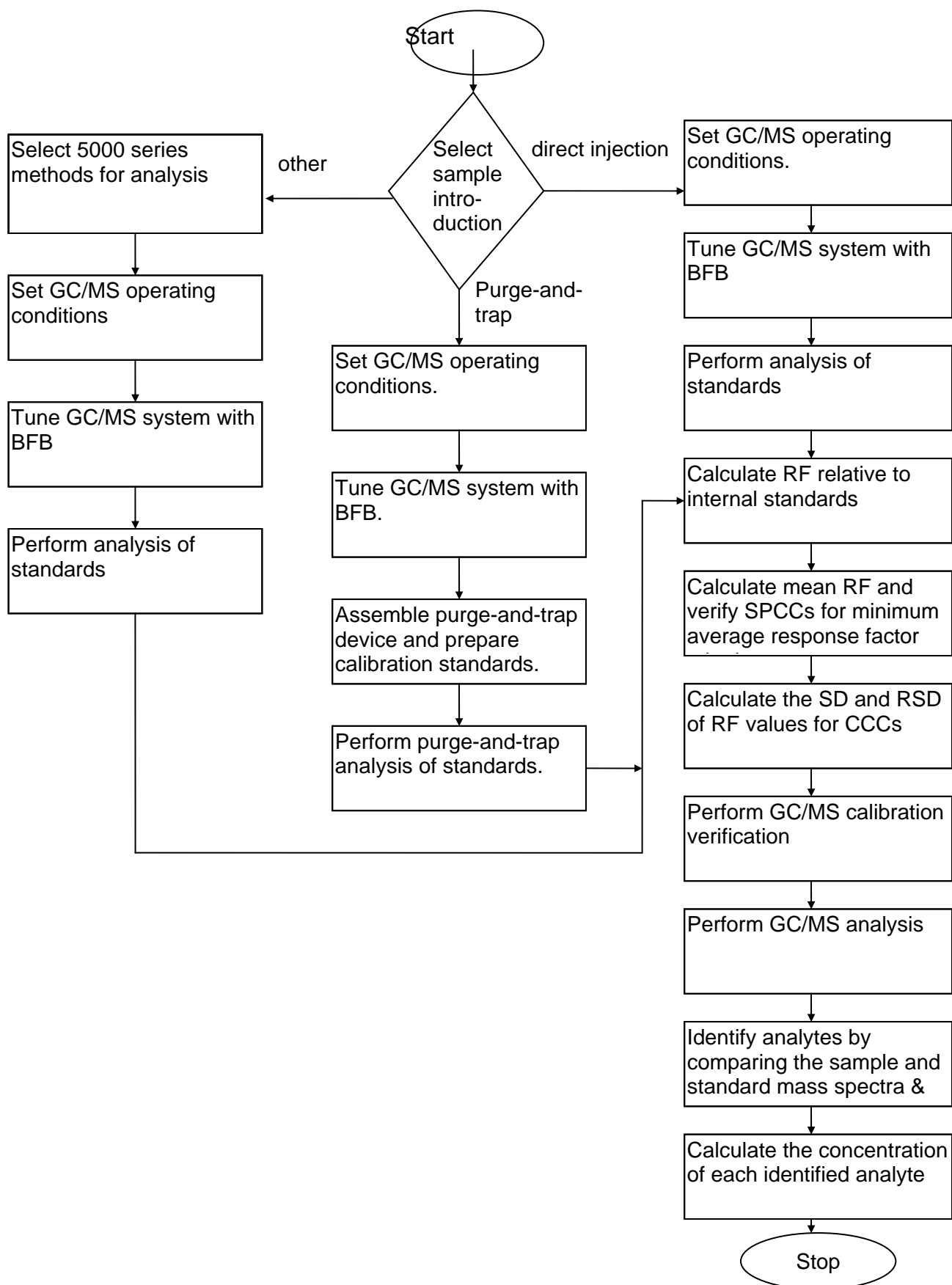
26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

- 26.1. Attachment I: Flow Chart
- 26.2. Attachment II: Client Specific Requirement Statement
- 26.3. Attachment III: Master Guide to Passing BFB for Agilent MSD Modes 5970-5973
- 26.4. Attachment IV: Agilent Document: BFB Tuning for Environmental Analysis: Three Ways to Succeed.
- 26.5. Attachment V: VOA Calibration Process
- 26.6. Attachment VI: VOA Calibration Checklist

27. Revisions

Document Number	Reason for Change	Date
S-GB-O-056-Rev.08	Section 2, 10.1: Added information for Nitrogen purge. Table 7.1: Updated samples to 3 vials. Table 9.1: Updated Serial Number for equipment, added 40MSVC. Tables 10.3, 10.4, 10.6, and 10.7: Updated standard information. Table 11.1: Added 1-Methylnaphthalene Section 12.4: Added Operation of the Software Systems Section 12.5.1.4: Incorporated label weight of 0.18g into determinations. Attachments V and VI: Added.	09May2014
S-GB-O-056-Rev.09	Table 7.1: Updated preservation requirement to $\leq 6^{\circ}\text{C}$ from $2\pm 4^{\circ}\text{C}$ to match 40CFR. Table(s) 10.6, 10.7, and 11.1: Added information for CAL-3 on the water curve. Table 10.7: Added CAL 7 Curve information. Table 13.1: Added ME requirements for SC requirements. Section 25: Added Pace QM and TNI references.	02Dec2014
S-GB-O-056-Rev.10	Table 9.1: Removed 40MSV9 from instrument list; removed serial numbers from SOP. Table 10.3: Updated Stock Standards. Table 10.4: Added compounds to O2Si custom mix (changed from Restek); Removed Restek custom mix; Added compounds to 4.1 Mega Mix.	19Aug2015
S-GB-O-056-Rev.11	Throughout Document: Updated Pace Analytical Services, Inc to Pace Analytical Services, LLC Section 14.2: Added Carry-over protocols.	21Jun2017

FLOWCHART



Attachment II:

Throughout document, reference to Client Specific requirements refers to samples analyzed following the BP Technical Requirements LaMP Revision 10.1, Canadian National Railway Services and Technical Specifications Manual.

Attachment III: Master Guide to Passing BFB for Agilent MSD Modes 5970-5973.

ENVIRONMENTAL CONSULTING AND SUPPLIES, INC.
2227 RT. 1- SUITE 301
NORTH BRUNSWICK, NJ 08902
☎ TEL: (800) 327-9559
① FAX: (732) 390-1079
🌐 Website: www.ECSMDL.com
GC/MS Consultants in the Standards Business



Master Guide to Passing BFB for Agilent MSD models 5970-5973

The Importance of Thermal Stabilization

Before we begin discussing tuning let me make an important point. If a GC/MS has been vented, it takes quite a while for the system to thermally stabilize. So, even if you reached your ultimate pressure and all heated zones are at their setpoint and the high vacuum pump is at its setpoint, don't be fooled into thinking the mass spec has thermally stabilized. Even though the temperatures are at their setpoints, it takes several hours for the heat to fully disseminate throughout the analyzer. You can certainly run the systems prior to thermal stabilization, but don't be surprised if the system changes while you do it. Use the following guide as to how long thermal stabilization should take:

<u>Model MSD</u>	<u>Typical time for thermal stabilization</u>
5970	16 hours
5971	8 hours
5972	8 hours
5973	8 hours

Creating and Maintaining an Optimized Manual Tune File for BFB

OK, so let's assume your system has reached thermal stabilization and has passed Autotune. You now want to manually tune the system for BFB. Here is the procedure I use to take an Autotune file and generate a Manual Tune File for the MSD systems for both 524.2 and 624/8260:

Set the Oven Temperature to the temperature at which BFB elutes in your program. Since sensitivity is flow dependent, and flow is temperature dependent, we want to tune the system under the *same* conditions that BFB will experience when it elutes. Generally this temperature is around 150°C, depending on your configuration.

Change the scan range from 10-800 or 10-600 AMUs (or whatever it is in AUTOTUNE) to 10-300 AMUs. Most EPA Methods for Volatiles specify a 35-300 AMU range so 10-300 is fine.

We want to scan in the 10-29 range so we can leak check as we tune. We don't want to scan above 300 amu when we tune for BFB because that's not what the method stipulates. Set the 3 ions that are monitored from 69-219-502 to 69-131-219.

Once you have verified that no leak exists (the kind of leak where air gets sucked into the system), you can set the scan range to 35-300 (or whatever you use in your acquisition method). This isn't necessary but some analysts find it helpful. If you do this, be sure to reset the scan range to 10-300 each time you retune to check for air leaks and if none exists set it back to 35-300 amu.

At this point you need to establish in your mind the target relative abundances of 131 and 219. A good starting point would be 35-40% of each relative to ion 69.

Set the X-ray lens to maximize on ion 131 (5970, 5971, 5972). Theoretically ion 3 ions should maximize at the same point but sometimes the ramp is a bit skewed.

Our next step is to lower 219 (which should be around 60% or so from Autotune) and bring it even with 131 and to about 35-40% of ion 69. Generally, 131 is about 20% lower than 219 in Autotune. Both of these can be achieved by doing the following:

raising the Ion Focus from at or near 0 (where it should be after Autotune) to about 30-80 Volts for 5970

raising the Entrance Lens Offset from below 8 (where it should be after Autotune) to about 15-20 for 5971, 5972, 5973.

Since raising the Ion Focus/Ent Lens Offset increases overall sensitivity, after step 4 is done we will need to *lower* the EM voltage to stay on scale. Generally, aim for between 2-4 million counts of ion 69 adjust the EM in to keep the same abundance throughout the tuning procedure.

Do Peak width and Mass Axis calibrations. At this point, you can use the automated feature whereby the software does it for you. Later on, we can tweak it using the AMU Gain and AMU Offset if necessary.

Make minor adjustments in the Entrance Lens (5970) and Entrance Lens and/or Ion Focus (5971,2,3) to fine tune your ratios. Additionally, you can make minor adjustments on the Repeller if needed to fine tune ratios, but only do this as a last resort.

Try to keep the Repeller setting constant and set to whatever it sets it to in AUTOTUNE.

Often you need to go back and forth between both lenses to zero in on your target ratios. Remember to adjust the EM voltage to maintain proper abundance of ion 69.

Do Peak width and Mass Axis calibrations. Your masses obtained in Spectrum Scan (*not* Profile Scan which tells you Peak Width, not Mass Axis) should be the integer ± 0.1 AMUs (i.e. 68.90-69.10 etc.). If this cannot be obtained automatically by the software, a hardware problem may exist.

Peak widths for all three ions should be between 0.47-0.54 AMUs, as close to 0.50 AMUs as possible (although in my experience 0.52 seems to be a little better). If they are not, they can be narrowed by increasing the AMU Gain and AMU offset and widened by decreasing either or both of these settings. If you cannot achieve ratios between 0.47-0.54 AMUs for all three ions, a hardware problem may exist.

For the 5971, 72 and 73s, you can also fine tune the width of ion 219 with the "219Wid" setting, although keep in mind that adjusting that also affects the 131 peak width as well.

Save the settings under a new name (or overwrite BFB.U if that file already exists) and run your BFB. It will usually pass. If not, the system must be re-tuned. Some systems require ion 131 to be greater than 219 (typically 40-35 or 35-30, etc.); others require ion 219 to be greater than 131 (typically 40-35 or 35-30, etc.). Adjustment of these ratios can be achieved by varying the Ion Focus and/or Entrance Lens Settings (especially the Entrance Lens). If you cannot pass BFB by having 131=219, or 131<219 by about 5-15%, or 131>219 by about 5-15%, this indicates that a problem exists. You should not have to obtain any weird abundances to make BFB pass. Generally, a system with 131 and 219 about the same abundance and both between 25-45% of 69 will pass. But since each system is unique and all will change with time and usage you must get a feel for what works best for your system.

Remember: once you have a good Tune File, you should check the tune each day and make whatever adjustments are necessary to keep ratios, abundances, and peak widths constant; this is fundamentally important in maintaining linearity and consistency in your analytical runs. You don't need to re-run Autotune each day- go directly to Manual Tune and adjust the system to look as it did the day before. Do this each day and you will be doing quite a bit to help your system stay linear.

So now we run our BFB and it will usually pass at the apex. But what if it doesn't? What if our relative ratios and peak widths of ions 69-131-219 are exactly what we think they should be and exactly what's been working for the last few months? What do we do then? We'll review the acceptance criteria for BFB (and since various methods have various criteria we will use the tightest acceptance criteria), what can fail, and how to modify your tune file to make BFB pass.

Remember: GC/MS systems are dynamic instruments: their sensitivities and responses change with time and usage, and what works today on your system may not work forever; it's essential that good GC/MS analysts understand tuning and be able to fine-tune (no pun intended) you systems to pass BFB and keep it running optimally.

Let's begin by assuming we have tuned our instrument to have all peak widths at or near 0.50+-0.05 amu, the mass axis of ions 69,131 and 219 are all +-0.1 amu, and the relative ratios of 69-131-219 are 100%-35%-35% respectively. We run our BFB and it fails. We then MUST make adjustments to our Manual Tune file based on what failed, correct our ratios and/or peak widths accordingly, and rerun BFB.

I will give some guidelines to follow in modifying your tune file. If these guidelines fail to make BFB pass, a hardware problem may exist.

The chart below lists the acceptance criteria for BFB we will use in this discussion. Keep in mind that some methods and stage agencies allow you to use the Apex, The Apex + 1 scan, The Apex + 1 scan or a 3-scan average of them so be sure you're trying all the legal scans in the peak. Using scans other than the Apex and one scan to either side should not be used. Also, if you have a significant baseline you should obtain a background subtracted mass spectrum.

Acceptance criteria for 4-Bromofluorobenzene for 624/8240

Source: EPA Method 624 for 50 ng injection

Mass	Acceptance Criteria	Affected by in Tune File
50	15-40% of mass 95	ratio of <u>69</u> to 131 and 219
75	30-60% of mass 95	ratio of <u>69</u> to 131 and 219
95	Base peak, 100% relative abundance	ratio of <u>131</u> to 69 and 219
96	5-9% of mass 95	peak width of ion <u>131</u>
173	<2% of mass 174	peak shape of ion <u>219</u>
174	50-99.9 of mass 95	ratio of <u>219</u> to 131 and 69
175	5-9 of mass 174	peak width of ion <u>219</u>
176	95-101% of mass 174	ratio of <u>219</u> to 131 and 69
177	5-9% of mass 176	peak width of ion <u>219</u>

As the chart illustrates, for every criteria of BFB there is a corresponding ion in the compound PFTBA (Perfluorotributylamine) which is used during tuning. So if the BFB fails for one or more criteria, we adjust the ratios and/or peak widths of the PFTBA during tuning.

If you are having problems with BFB, ALWAYS check your high vacuum pressure. The discussion that follows assumes that your ion source pressure is consistent with what it historically has been. If not, that needs to be resolved first and foremost before any of the techniques presented here can be utilized.

We will now discuss each ion and what to modify in the Manual Tune file should it fail:

Ion 50 (Acceptance criteria: 15-40% of mass 95); Affected mainly by the ratio of 69 to 131 and 219 in our tune file. Tests for adequate low-end sensitivity.

Tuning issues that cause problems with this ion:

Generally, if ion 50 fails it is because it falls under the 15% minimum percentage criterion. Occasionally it will fail because it is >40% of mass 95. I have seen many instances where ion 50 ends up below 15% of mass 95. This means that the system is not sensitive enough at the low end. To remedy this, lower the relative ratios of 131 and 219 each by about 5%. This reduction of the mid-range ends up making the low end more sensitive and will boost the amount of ion 50 that is generated. If this fails, continue to lower the relative ratios of 131 and 219. If you need to make ions 131 and 219 below 20% of ion 69 for BFB to pass I would suspect a hardware problem might exist.

If ion 75 ends up being too high (greater than 40% of ion 95), you should try raising both ions 131 and 219. This increase in the mid-range ends up making the low end less sensitive and should reduce the amount of ion 50 that is generated. If this fails, continue to raise the relative ratios of 131 and 219. If you need to make ions 131 and 219 above 50% of ion 69 for BFB to pass I would suspect a hardware problem might exist.

Hardware issues that cause problems with this ion:

The most common hardware issues that cause failure of ion 50 would be a dirty source (especially if you have elevated amounts of ion 50 AND elevated amounts of ion 75), problems with the rough pump (also for elevated amounts of ion 50) or a low-mass gain Electron Multiplier (for lower amounts of ion 50). A dirty source is remedied by cleaning the ion source (and replacing the filaments). Problems with the rough pump can be resolved by changing the rough pump oil (be sure only to use Inland 45 oil) and/or replacing the beads in the molecular sieve filter on the rough pump (for Oil Diffusion Pump systems). If neither of these works, it's possible that the rough pump may need replacement.

Ion 75 (Acceptance criteria: 30-60% of mass 95); Affected mainly by the ratio of 69 to 131 and 219 in our tune file. Like ion 50, tests for adequate low-end sensitivity.

Tuning issues that cause problems with this ion:

Generally, if ion 75 fails it is because it exceeds the 60% maximum percentage criterion. I have never seen it fail because it is <30% of mass 95. Sometimes ion 75 does end up above 60% of mass 95. This means that either the system is too sensitive at the low end or an indication that the source is getting dirty. To remedy this, first raise the relative ratios of 131 and 219 each by about 5%. This increase of the mid-range ends up making the low end less sensitive and will lower the amount of ion 75 that is generated. If this fails, continue to boost the relative ratio of 131 compared to 219. If this fails, try cleaning the ion source. If you need to make ion 131 and/or 219 above 60% for BFB to pass I would suspect a hardware problem exist.

Hardware issues that cause problems with this ion:

The most common hardware issues that cause failure of ion 75 would be a dirty source (especially if you have elevated amounts of ion 50 AND elevated amounts of ion 75), problems with the rough pump (also for elevated amounts of ion 75). A dirty source is remedied by cleaning the ion source (and replacing the filaments). Problems with the rough pump can be resolved by changing the rough pump oil (be sure only to use Inland 45 oil) and/or replacing the beads in the molecular sieve filter on the rough pump (for Oil Diffusion Pump systems). If neither of these work, it's possible that the rough pump may need replacement.

Note for EPA Method 524.2

2A. Ion 75 (Acceptance criteria: 30-80% of mass 95); Affected mainly by the ratio of 69 to 131 and 219 in our tune file. Like ion 50, it tests for adequate low-end sensitivity.

The EPA, in its infinite wisdom, widened the range for Ion 75 for method 524.2. I guess they figured since they're making you shoot a smaller amount of BFB (25 ng as opposed to 50 ng with 624/8260), they'll cut you some slack with the problematic ion 75. An upper limit of 80% makes it such that the source would have to be VERY dirty or some hardware problem would have to exist for it to fail.

Ion 95 (Acceptance criteria: Base peak, 100% relative abundance); Affected mainly by the ratio of 131 to 69 and 219 in our tune file.

Tuning issues that cause problems with this ion:

Generally, if ion 95 fails it is because ion 174 or 176 is the base peak. This means that ion 131 is too low and ion 219 is too high. To remedy this, raise the relative ratio of 131 and lower the relative ratio of 219 each by about 5%. This change will lower the 174/176 pair and should restore 95 to base peak status. If you need to make 131 greater than 219 by more than 15% for BFB to pass I would suspect a hardware problem might exist.

Hardware issues that cause problems with this ion:

If ion 95 fails it is because ion 174 or 176 is the base peak then the system might be running at below-ideal temperatures. In order to obtain proper ratios, the source and analyzer temperatures have to be correct. Be sure the system has thermally stabilized by allowing sufficient time (see discussion earlier in this issue). If the system has had enough time to thermally stabilize, then perhaps the analyzer is too cold. Use the following chart as a guideline:

MSD	Source temp	Quad temp	Transfer line temp
5970	200°C	same as source	250°C
5971	NA	NA	280°C
5972	NA	NA	280°C
5973	200°C to 230°C	150°C	280°C

Ion 96 (Acceptance criteria: 5-9% of mass 95); Affected mainly by the peak width of ions 69 and/or 131 in our tune file.

Tuning issues that cause problems with this ion:

Generally, if ion 96 fails it is because the peak width of ions 69 and/or 131 are not close enough to 0.50 amu. I have seen this isotope ion fail on both ends of the spectrum. If ion 96 falls below the 5% minimum, try narrowing the peak widths of ions 69 and/or 131. If ion 96 falls above the 9% maximum try widening the peak width of ions 69 and/or 131. Also, try lowering the abundance threshold and/or raising the Electron Multiplier setting.

Keep in mind that failure of this minor ion is often linked to poor peak shape in manual tune. So, even if the peak width is correct, you can still fail if the peak shapes of ions 69 and/or 131 are poor. I have seen many instances where elevated Entrance Lens settings distort the peak shape in Manual Tune. Sometimes, but not always, Entrance Lens settings above 100 mV/amu can cause distortion with the 5970, 71 and 72 and settings as low as 40 mV/amu can cause distortion with the 5973.

If this doesn't work, try changing the DC Polarity. For the 5970, this would be a small service issue as to do this one needs to swap 2 wires on the RFPA Board of the analyzer. For the 5971, 72 and 73, you can swap polarities in Manual Tune from POS to NEG (or vice versa). If you change Polarity, you'll need to do a peak width and mass axis calibration and retune the system as undoubtedly the ratios of 69-131-219 will change as well.

Hardware issues that cause problems with this ion:

If this issue can't be resolved by adjusting peak widths or changing polarities, a contaminated quadrupole and/or faulty electronics problem may exist. Usually, manual tune peak shape and corresponding isotope ratios are linked to problems with the RFPA (Radio Frequency Power Amplifier) electronics, so that would be the first thing to check.

You may also need to re-tune the RF coils to improve peak shape. This would be a service issue.

Ion 73 (Acceptance criteria: <2% of mass 174); Affected mainly by the peak shape of ion 219 in our tune file.

Tuning issues that cause problems with this ion:

This is an interesting one. Ion 173 should be absent or present in very small abundance. If it is found above the 2% of mass 174 level, it is usually because of poor peak shape in ion 219. Even if the peak widths are fine, ion 173 will fail if fronting occurs in ion 219 in Profile Scan. When tuning, you need to closely examine the peak shapes of all three ions, especially ion 219. If the peak shape of 219 is not Gaussian (symmetrical), ion 173 will be created at unacceptably high levels.

You can also try swapping the Polarity to see if peak shape improves. Also, be sure you have the Threshold set correctly in your acquisition method. It's possible that by raising the Threshold you may remedy this problem.

Hardware issues that cause problems with this ion:

The first thing to do is to clean the source, paying extra close attention to the Entrance Lens. The Entrance Lens is the component of the Ion Source that comes in contact with the quadrupole, so contamination on the Entrance Lens can affect peak shape. For you 5970 users, it would be a good idea to swap Entrance Lenses if the white ceramic insulator looks excessively dirty.

Another possible remedy would be to clean the quadrupoles. (Warning: cleaning the quadrupoles is NOT considered routine maintenance as is cleaning the source and should only be done by trained personnel.)

Ion 174 (Acceptance criteria: 50-99% of mass 95); Affected mainly by the abundance of ion 219 relative to 69 and 131 in our tune file.

Tuning issues that cause problems with this ion:

I have seen this ion fail on both ends of the spectrum. If ion 174 is too large (i.e. it's the base peak), lower the relative abundances of both 131 and 219, especially ion 219. Try setting 131 about 5-10% greater than 219 in you tune file. Conversely, if ion 174 falls below 50% of mass 95, raise the relative abundances of both 131 and 219, especially ion 219. Try setting 219 about 5-10% greater than 131 in you tune file. Refer to the discussion a few pages back regarding ion 95 being the base peak.

Hardware issues that cause problems with this ion:

If ion 174 or 176 is the base peak then the system might be running at below-ideal temperatures. In order to obtain proper ratios, the source and analyzer temperatures have to be correct. Be sure the system has thermally stabilized by allowing sufficient time (see discussion earlier in this issue). If they system has had enough time to thermally stabilize, then perhaps the analyzer is too cold.

Tuning issues that cause problems with this ion:

This is a similar situation to ion 96, only ion 219 in our tune file is the key ion as opposed to 69 and/or 131. Generally, if ion 175 fails it is because the peak width of ion 219 is not close enough to 0.50 amu. I have seen this isotope ion fail on both ends of the spectrum. If ion 175 falls outside the 5-9% window, try widening or narrowing the peak width of ion 219. Try setting the peak width to 0.45 amu, then 0.50 amu, then 0.55 amu and finally 0.60 amu. You can accomplish this by raising or lowering the AMU gain and/or AMU offset. Also, adjusting the 219 Wid setting (for 5971, 2 and 3 MSDS) also can be adjusted. It's dangerous to set the peak widths much further from the 0.45-0.60 as this can cause the mass spec to mis-assign masses (i.e. it will not be able to reliably resolve one mass from another.)

You can also try swapping polarities.

If ion 175 is absent in some of the scans, you can also try changing the A/D setting (usually raising the setting helps this problem). Details of this are given in the next section.

If none of this works, faulty electronics and/or a contaminated quadrupole may exist.

Hardware issues that cause problems with this ion:

First, try cleaning the ion source.

Another possible remedy would be to clean the quadrupoles. (Warning: cleaning the quadrupoles is NOT considered routine maintenance as is cleaning the source and should only be done by trained personnel.)

Also, the rough pump plays a role in properly resolving this ion. Check the foreline pressure. For oil diffusion pump systems like the 5971, 5972 and some 5973s, this is reported on your Manual Tune report as Vacuum (expressed as millitorr). Foreline pressures above 60 mtorr can cause problems with the mass spec being unable to resolve Ion 75.

Ion 176 (Acceptance criteria: 95-101% of mass 174); Affected mainly by the abundance of ion 219 relative to 69 and 131 in our tune file.

Tuning issues that cause problems with this ion:

I have seen this ion fail on both ends of the spectrum. The problem is that there is no known remedy as far as adjustment of ratios or peak widths. The usual solution is to perform a 3-scan Enhancement (i.e. averaging of the Apex + -1 scan) or try either the Apex-1 or the Apex +1 scan. Often times a passing spectrum will result. If this happens only occasionally, then it was probably a fluke and I would just shoot BFB again-it'll probably pass. If it's a chronic problem, double check you A/D (Analog to Digital) setting (Also called Sampling rate on some systems). You might want to *increase* the A/D setting such that each scan on your peak is an average of more scans and is a more accurate representation of the true spectrum. Usually, megabore columns employ A/D of $2^3=8$. Try $2^4=16$ and see if the problem is solved. If you are using $2^2=4$, try using $2^3=8$ for your A/D.

Hardware issues that cause problems with this ion:

Often times, cleaning the Ion source will remedy this problem.

Ion 177 (Acceptance criteria: 5-9% of mass 176); Affected mainly by the peak width of ion 219 in our tune file.

Tuning issues that cause problems with this ion:

This is a similar situation to ion 175, although this ion is a lot less problematic. Generally, if ion 177 fails it is because the peak width of ion 219 is not close enough to 0.50 amu. I have seen this isotope ion fail on both ends of the spectrum. If ion 177 falls outside the 5-9% window, try widening or narrowing the peak width of ion 219. Try setting the peak width to 0.45 amu, then 0.50 amu, then 0.55 amu and finally 0.60 amu. You can accomplish this by raising or lowering the AMU gain and/or AMU offset. Also, adjusting the 219 Wid setting (for 5971, 2 and 3 MSDS) also can be adjusted. It's dangerous to set the peak widths much further from the 0.45-0.60 as this can cause the mass spec to mis-assign masses (i.e. it will not be able to reliably resolve one mass from another).

You can also try swapping polarities.

If none of this works, faulty electronics and/or a contaminated quadrupole may exist.

Hardware issues that cause problems with this ion:

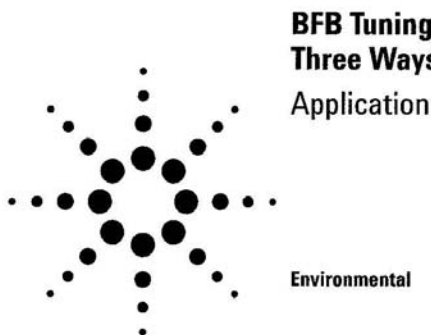
First, try cleaning the ion source.

Another possible remedy would be to clean the quadrupoles. (Warning: cleaning the quadrupoles is NOT considered routine maintenance as is cleaning the source and should only be done by trained personnel.)

Summary of BFB Tuning

Usually ratios of 69-131-219 of 100%-37%-37% respectively and peak widths at 0.50 amu will cause BFB to pass...but NOT ALWAYS. Keep in mind that you need to make whatever adjustments are necessary to make BFB pass. Volatile systems are less dynamic that Semivolatile systems generally because the source stays cleaner (less contamination hits the detector on a purged sample than in a Methylene Chloride extract) so the drift is less frequent and less severe. But all GC/MS systems eventually show some change.

Attachment IV: Agilent Document: BFB Tuning for Environmental Analysis: Three Ways to Succeed.



Author

Philip L. Wylie
Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19808-1610
USA

John E. Pellerin
Agilent Technologies, Inc.
40 Shattuck Road
Andover, MA 01810
USA

Abstract

The United States Environmental Protection Agency methods 524.2, 8260B, and Contract Laboratory Program Statement of Work employ purge and trap concentration of volatile compounds in water samples with analysis by gas chromatography/mass spectrometry. Each method requires the mass spectrometer to meet specific tuning criteria before proceeding to actual samples. This paper summarizes these tuning criteria, and shows three different ways that the Agilent Technologies 6890/5973 gas chromatograph/mass selective detector system can be tuned to meet them. A very simple and robust procedure is described in the Modified Autotune section. A quick reference guide for this procedure is given at the end of the paper under Modified Autotune Summary.

Introduction

If you are already familiar with 4-bromofluorobenzene (BFB) tuning and evaluation procedures, you may want to go directly to the section titled "Modified Autotune Summary" found at the end of this paper. It offers an alternative approach for tuning Agilent 6890/5973 GC/MSD systems that is routinely successful in this laboratory.

The United States Environmental Protection Agency (USEPA) has developed several methods for the analysis of volatile organic compounds (VOCs) in water samples. The three most widely used procedures all employ purge and trap (P&T) sample introduction followed by capillary column gas chromatography with mass spectral detection (P&T/GC/MS). USEPA Method 524.2 revision 4¹ is used for drinking water analysis while Method 8260B revision 2² is used for wastewater. The USEPA Contract Laboratory Program Statement of Work (CLP-SOW)³ uses a similar P&T/GC/MS method for the analysis of hazardous waste.

There are many similarities among these three USEPA volatiles methods. One common requirement is that the GC/MS system must be tuned in such a way that 4-bromofluorobenzene (BFB) meets specific ion abundance criteria. This requirement helps to ensure that data are comparable between instruments of different design and

among various laboratories. This paper summarizes USEPA method 524.2, 8260B, and CLP tuning criteria, and shows three different ways that the Agilent Technologies 6890/5973 GC/MSD system can be tuned to meet them.

Experimental

A standard containing fluorobenzene, 1,2-dichlorobenzene- d_4 , and 4-bromofluorobenzene at 2.0 mg/mL was purchased from AccuStandard (New Haven, CT). A portion of this solution was diluted in methanol (B&J HPLC and pesticide grade) to a concentration of 50 ng/ μ L.

Standards for tune evaluation were injected by syringe or P&T into several different Agilent Technologies 6890/5973 GC/MS systems. When making syringe injections into the split/splitless inlet, a liner with a 900- μ L volume was used and no more than 1.0 μ L was injected to avoid over-expansion in the inlet.

Results and Discussion

Tuning Criteria

Table 1 lists the tuning criteria for USEPA methods 524.2, 8260B, and CLP-SOW. All three methods base their tuning criteria on the ion responses of BFB. All ion responses are reported relative to m/z 95, which is assumed to be the base

peak even though ions 174 and 176 may be larger in the CLP-SOW method.

While many of the requirements in Table 1 are the same for all three methods, some important differences are worth noting. Method 8260B actually allows the analyst to use the tuning criteria specified in either of the other two methods. More importantly, it allows one to use "manufacturers tuning (sic) instructions" so long as it does not hurt method performance. However, many laboratories still follow the BFB tuning requirements specified in method 8260B or choose to substitute CLP-SOW tuning criteria.

Methods 524.2 and 8260 require that m/z 95 be the base peak in the BFB spectrum, which caps the m/z 174 relative abundance at 100% (relative to m/z 95). The CLP-SOW requirements allow m/z 174 to be up to 120% of m/z 95. Tuning procedures that reduce the response of m/z 174 too much may lead to lower overall sensitivity, especially for bromoform which has a quant ion of m/z 173. Conversely, maximizing this ratio, within the requirements of the method, can enhance overall sensitivity.

Automated BFB Tuning

The Agilent 5973 MSD uses perfluorotributylamine (PFTBA) for electron impact tuning because it exhibits good stability, the right volatility, and a wide range of fragment masses. However, USEPA volatiles methods evaluate the tune using BFB which produces an entirely different spectrum.

Table 1. Criteria for BFB Tuning for Three Capillary GC/MS Volatiles Methods

Mass (m/z)	Relative Abundance Criteria		
	Method 524.2	Method 8260B ^a	CLP-SOW
50	15 to 40% of 95	Same as 524.2	8 to 40% of 95
75	30 to 80% of 95	30 to 60% of 95	30 to 66 % of 95
95	Base Peak, 100%	Same as 524.2	Same as 524.2
96	5 to 9% of 95	Same as 524.2	Same as 524.2
173	<2% of 174	Same as 524.2	Same as 524.2
174	>50% of 95	Same as 524.2	50 to 120% of 95
175	5 to 9% of 174	Same as 524.2	4 to 9% of 174
176	>95 to <101% of 174	Same as 524.2	93 to 101% of 174
177	5 to 9% of 176	Same as 524.2	Same as 524.2

^aAlternative tuning criteria may be used (for example, CLP or Method 524.2) including manufacturer's instructions provided that method performance is not adversely affected.

Therefore, automated (or manual) tuning procedures must adjust PFTBA ion responses in order to get the desired BFB response ratios. Agilent G1701CA EnviroQuant ChemStation software automates BFB tuning so that the instrument typically passes the more restrictive USEPA Method 524.2 and 8260B requirements listed in Table 1. After tuning, the analyst must inject a BFB standard by syringe or P&T to verify that the tune passes the requirements for the method in use.

Automated BFB tuning adjusts MSD source parameters so that PFTBA ion abundances meet predetermined "targets." The default PFTBA target values are set so that a subsequent BFB injection should meet the requirements for all three methods. Table 2 shows a portion of a BFB tune report that includes the target responses (as a percentage of m/z 69) for m/z 50, 69, 131, 219, 414, and 502. The actual abundances achieved by the tune are shown on the last line. When these targets

Table 2. A Portion of a Typical BFB Tune Report

Target Mass:	50	69	131	219	414	502
Target Abund (%)	1.0	100.0	45.0	55.0	2.4	2.0
Actual Tune Abund (%)	1.2	100.0	48.1	59.3	2.7	2.3

are met, the Agilent 5973 MSD normally passes any of the tuning criteria listed in Table 1.

Figure 1 shows an average spectrum obtained for a 1- μ L manual injection of BFB (50 ng/ μ L split 50:1) using the tune shown in Table 2. Agilent G1701CA EnviroQuant ChemStation Environmental Data Analysis software can evaluate the spectrum automatically and generate a report that is archived with the data file. Because BFB tuning criteria are not uniform among USEPA methods, the analyst must first specify the allowable ranges using the form shown in Figure 2. The form is accessed in Environmental Data Analysis by selecting Tuner/Edit BFB Criteria on the dropdown menu.

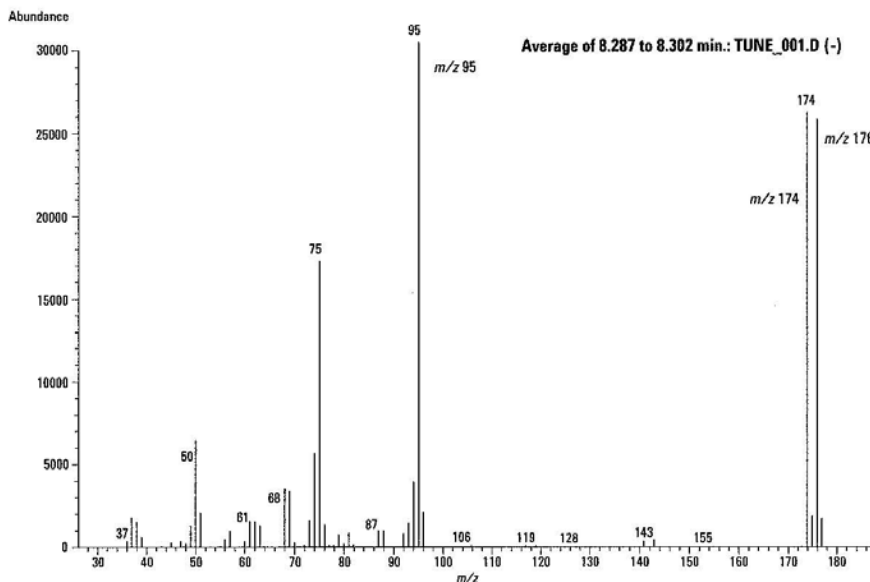


Figure 1. Average spectrum of BFB after performing a standard BFB automated target tune. One μ L of a methanol solution containing 50 ng/ μ L of BFB was injected by hand.

Tune Evaluation Criteria

Method Title: BFB

Base MZ: 95.00

Alternate Base MZ: 1.00

☐ Include Tabular Report With Printout

☐ APR Base OK

Mass	Rel. to MZ	% Low	% High	APR Base OK
50.00	95.00	15.00	40.00	N
75.00	95.00	30.00	60.00	N
95.00	95.00	100.00	100.00	N
96.00	95.00	5.00	9.00	N
173.00	174.00	0.00	2.00	N
174.00	95.00	50.00	100.00	N

50.00 95.00 15.00 40.00

Enter Delete Report OK Cancel Help

Figure 2. The Agilent G1701CA EnviroQuant ChemStation screen for entering BFB tune criteria. The user can modify the parameters to meet the requirements of the method in use. These values are used by the ChemStation for automated tune evaluation.

Having entered abundance criteria for the method in use, one can automatically assess the suitability of the tune using the EnviroQuant software (Figure 3). One can choose to "Evaluate BFB to Screen/Printer" in which case it will evaluate the current spectrum. This can be a single spectrum or an average. Alternatively, by choosing "Autofind BFB to Screen/Printer," the software automatically finds BFB in the chromatogram, averages the top three spectra and subtracts a baseline spectrum. In either case, a report such as the one in Figure 4 is generated. The most recent report is archived in the datafile.d directory in a file called tuneeval.txt.

Select BFB Evaluation

☒ Evaluate BFB to Screen

☐ Evaluate BFB to Printer

☐ Autofind BFB to Screen

☐ Autofind BFB to Printer

OK Cancel Help

Figure 3. Choices for automated BFB tune evaluation by the EnviroQuant software. The "Evaluate BFB..." choices use the spectrum (single or averaged) in Data Analysis window 1 for evaluation. The "Autofind..." choices automatically find the BFB peak, average the top three BFB spectra and subtract a baseline spectrum prior to evaluation.

BFB

Data File : C:\MSDCHEM\1\DATA\Sep24_01\TUNE_001.D Vial: 1

Acq On : 24 Sep 2001 2:25 pm Operator:

Sample : BFB for tuning 1 uL Inst: GC/MS Ins

Misc : Multiplr: 1.00

MS Integration Params: rsteint.p

Method : C:\MSDCHEM\1\methods\envdef.m (RTE Integrator)

Title :

AutoFind: Scans 1567, 1568, 1569; Background Corrected with Scan 1559

Target	Rel. to	Lower	Upper	Rel.	Raw	Result
Mass	Mass	Limit	Limit	Abn	Abn	Pass/Fail
50	95	15	40	21.6	6769	PASS
75	95	30	60	56.5	17708	PASS
95	95	100	100	100.0	31317	PASS
96	95	5	9	7.3	2209	PASS
173	174	0.00	2	0.0	0	PASS
174	95	50	100	82.8	25933	PASS
175	174	5	9	7.4	1910	PASS
176	174	95	101	99.3	25747	PASS
177	176	5	9	6.6	1702	PASS

TUNE_001.D envdef.m Mon Sep 24 16:00:27 2001 MSVOC04

Figure 4. The Agilent EnviroQuant ChemStation BFB Tune Evaluation Report for the spectrum shown in Figure 1.

In this case the automated BFB tuning procedure produced a tune that passes Method 524.2 and 8260B criteria with a 174/95 ratio of 82.8%. This ratio is limited to 100% by these USEPA methods, which specify that m/z 95 must be the base peak. To meet these strict guidelines, one has to "de-tune" the Agilent 5973 MSD which results in somewhat lower instrument sensitivity. Laboratories may want to increase the 174/95 ratio so it more closely approaches the 100% limit of Methods 524.2 and 8260B or so that it approaches the 120% limit specified in the CLP-SOW method. Most laboratories that perform Method 8260B tune their instruments to meet the CLP-SOW requirements because the method allows laboratories to use these tune criteria and the MSD performance is closer to optimum.

In addition to the automated BFB tune, there are two procedures that can be used to improve instrument sensitivity, to meet the more liberal CLP-SOW requirements, or to create a passing tune should the standard BFB autotune fail. In this laboratory, the "Modified Autotune" procedure was found to produce tunes that routinely passed BFB criteria for any of the three methods. As shown below, changing the BFB tuning targets can also produce a passing BFB tune while enhancing the signal for bromoform.

Target Tuning

Automated BFB tuning adjusts MSD source parameters to achieve the target responses required for the method in use. This is essentially a "target tune" procedure where the initial target abundances provided by the software are designed to

meet the more restrictive 524.2 and 8260B requirements. When needed, it is easy to change the target PFTBA relative abundance criteria to produce the desired affect on the BFB ions. This is done by selecting View/Manual Tune/Set Tune Targets.

For example, consider the spectrum in Figure 1 which passed all of the tuning criteria, but which had a lower than optimum m/z 174 response. Experience in this laboratory has shown that increasing the relative abundance of m/z 174 will increase the overall sensitivity of the instrument, in particular for the bromoform response at m/z 173. As shown in Figure 5, the target abundances for ions 131 and 219 were each increased to 70% from their default values of 45% and 55% respectively. These choices were saved to the BFB.U tune file and a new BFB Target Tune was run. Figure 6 shows the new BFB spectrum (average of three spectra across the apex with baseline subtraction) which passes CLP-SOW criteria (Table 1) and is, therefore, satisfactory for either CLP or 8260B volatiles methods.

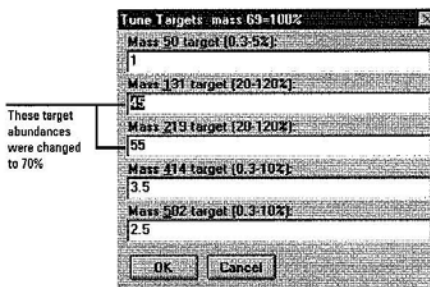


Figure 5. PFTBA target abundance values (relative to m/z 69) used for "target" tuning. When these abundances are saved to the BFB.U tune file, they are used by the BFB target tune algorithm.

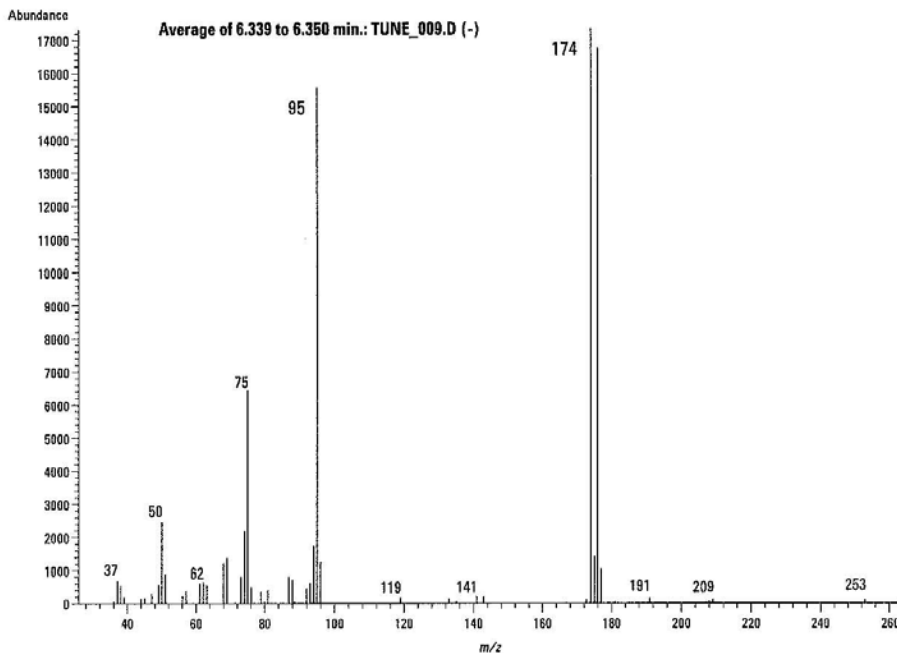


Figure 6. Average BFB spectrum obtained by changing the tune targets for m/z 131 and 219 to 70% (relative to m/z 69). This spectrum passes CLP-SOW tuning criteria.

Modified Autotune

With the convenience of automated tuning procedures available in the Agilent ChemStation software, most analysts have gladly given up the idea of manually tuning their 5973 MSDs. A combination of automated tuning with a slight manual modification has given excellent BFB results in this laboratory. The total process is easy and usually takes just a few extra minutes after the autotune is complete. The steps are described below and are summarized in a "quick reference" format in the next section.

1. From the Manual Tune portion of the software, perform an Autotune (select Tune/Autotune). This algorithm tunes the Agilent 5973 MSD for maximum sensitivity over the entire mass range and is widely used by methods that do not specify other tune criteria. This autotune emphasizes overall sensitivity by improving abundances for higher mass ions (for example, 502). As a result, the Autotune procedure typically gives an abundance for m/z 50 that is too low to meet 524.2 and 8260 criteria and an abundance of m/z 174 that may be too high, even for CLP-SOW tuning.
2. After completing the Autotune procedure, choose Edit MS Params (under the AdjParam menu item) which will display the screen shown in Figure 7.

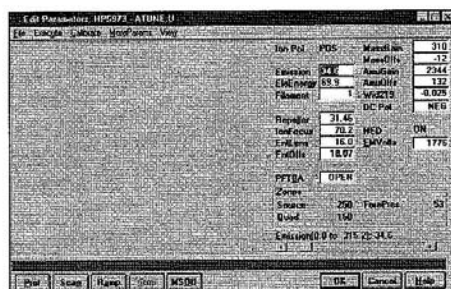
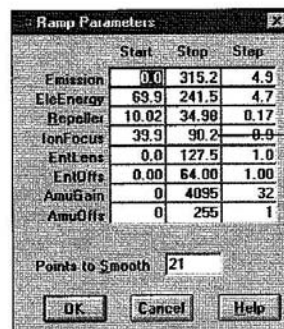


Figure 7. The Edit Parameters screen found by selecting AdjParam/Edit MS Params in the main Manual Tune window.

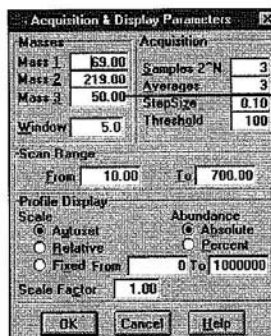
3. Two changes are required in the default values used for adjusting parameters in this view. First, under the MoreParams menu, choose Ramp Params and change the "Stop" value for the ion focus to 140 as shown in Figure 8. Close this window and choose

AcqParams under the MoreParams window and change Mass 3 from 502 to ion 50 as shown in Figure 9. Close this window and return to the main Edit Parameters screen (Figure 7).



Change the IonFocus "Stop" value to 140

Figure 8. This window allows the user to set ranges for the various tuning parameters. The default ion focus "Stop" setpoint of 90 was set to 140.



Mass 3 has been changed from the default value of 502 to 50

Figure 9. Acquisition and Display Parameters window. M/z values of 69, 219, and 50 have been chosen so that these responses can be ramped and their relative abundances displayed.

4. Highlight the IonFocus window with the cursor and then select Ramp. This gradually ramps the ion focus voltage over the specified range while monitoring the response of ions 69, 219, and 50. After about a minute, a plot of these ion responses vs. the ion focus voltage appears in the window (Figure 10).

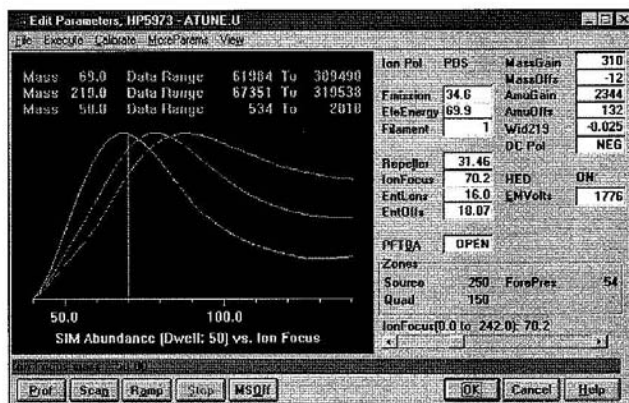


Figure 10. Abundances for ions 69, 219, and 50 while ramping the Ion Focus from 40 to 140.

- Under the View dropdown menu item, choose Expand. This view shows the current Ion Focus setting, the abundance of m/z 69 and the relative abundances of ions 219 and 50 (Figure 11). From the plot, it is easy to see that an increase in the Ion Focus value should increase the 50:69 ratio while reducing the 219:69 ratio. These are

exactly the changes that should enable the MSD to pass BFB tuning criteria.

Note that the ion focus ramping procedure can also be performed from the main Manual Tune screen by choosing Ramp/Ramp Ion Focus on the dropdown menu.

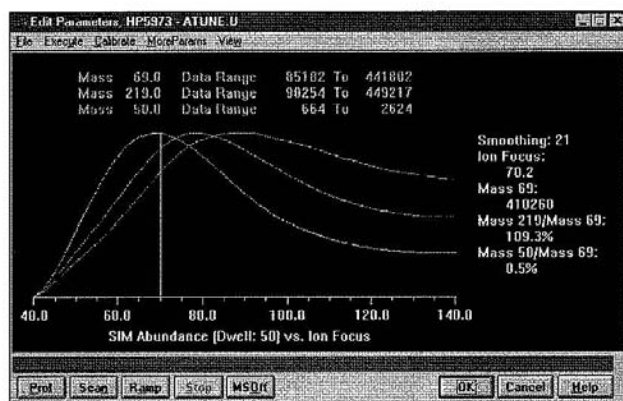


Figure 11. An expanded view of the SIM-Abundance-vs-Ion Focus plot obtained by selecting View/Expand. This view allows one to drag the vertical line to different setpoints while observing changes in the ion relative abundances.

6. The vertical line indicates the current ion focus setpoint. Use the cursor to drag this setpoint line to the right while observing the change in the 219:69 and 50:69 ratios. Agilent laboratories have had good success by setting the Ion Focus to values between 100 and 135 V. This should result in a 219:69 ratio in the 60-80% range and a 50:69 ratio that is 0.8 or greater. If tuning to meet 524.2 requirements, the 219:69 ratio should be on the low side of this range.

An alternative to the above procedure is to select Scan in the Edit Parameters window (Figure 7) while monitoring ions 69, 219, and 50. The 219:69 and 50:69 ratios are displayed under the Relative Abundance heading and are updated with each scan. Highlight the Ion Focus setting and adjust its value using the slider bar. The effect of different Ion Focus values will be seen almost immediately in the ion ratios. These ratios will bounce around somewhat, but trends can be seen over a few scans. A good choice for the 50:69 ratio would be about 0.85.

7. Click OK and return to the Manual Tune screen. Under the Calibrate menu item, choose Adjust Abundances, which will automatically reset the electron multiplier to get ion abundances in the optimum range. Save the tune, choosing a new name for the tune file (for example, BFB1.U). Return to Instrument Control (View/Instrument Control) and be sure to select this tune file for the method used to acquire the BFB checkout chromatogram. Inject or purge an appropriate amount of BFB and evaluate the tune using the software tools provided (Figures 2 through 4). Assuming that it passes, assign this tune to the P&T/GC/MS volatiles method in use.

Figure 12 shows the spectrum (average of the three scans across the apex with baseline subtraction) for a 1- μ L syringe injection (50 ng/ μ L split 50:1) of BFB using an ion focus value of 115 V. All other parameters (except for the electron multiplier) were set by the Autotune algorithm. This spectrum passes any of the tuning criteria listed in Table 1 but has a higher 174/95 ratio than was achieved using the standard BFB tune.

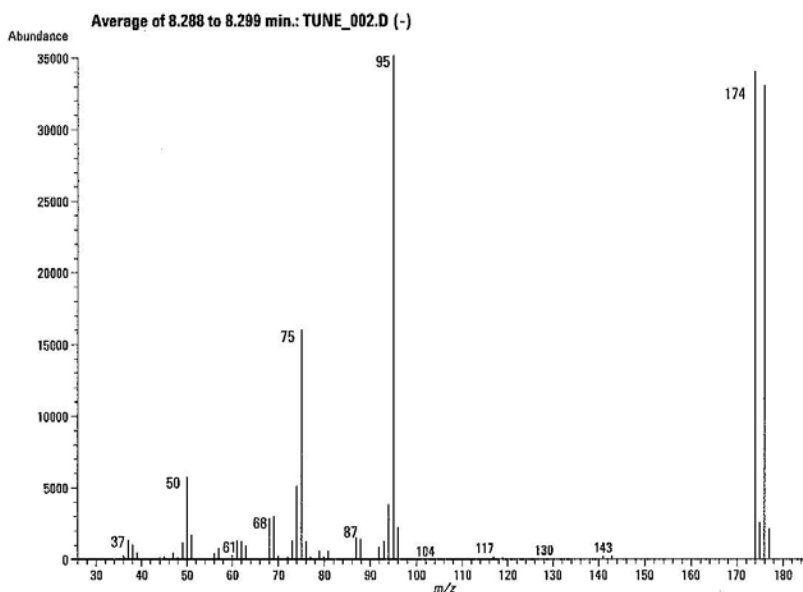


Figure 12. Average spectrum of BFB obtained after using the procedure described under Modified Autotune. After running a standard Autotune, the Ion Focus value was increased to 115 V.

The true test of a successful BFB tune is whether it holds up during repetitive VOC analyses and through normal instrument maintenance procedures. In one extreme test, the same BFB tune easily passed CLP-SOW criteria during a period when two different MSD sources were installed and four different filaments were used. On one Agilent 6890/5973 GC/MS instrument this procedure did not work until the MSD source was cleaned.

Finally, a note of caution is appropriate. While these techniques have worked well for the Agilent 6890/5973A and N GC/MSD systems, this does not imply that the same procedures are appropriate for older Agilent MSDs. Tuning frequency is dictated by the nature of the samples, choice of column and other factors such as column bleed and source cleanliness. If the source becomes too dirty, it must be cleaned in order to pass BFB tuning criteria, no matter which approach is taken.

Modified Autotune Summary

These steps summarize the procedure for modifying the standard Agilent 5973 Autotune to pass BFB tuning criteria. It is provided here as a quick reference guide for those who are already familiar with tuning procedures.

1. In the Manual Tune portion of the Agilent GC/MS ChemStation software, perform a standard Autotune.
2. In the Ramp Parameters window, change the Ion Focus Stop value to 140.
3. In the Acquisition & Display Parameters window, change ion 502 to 50.
4. In the Edit Parameters window click on Ion Focus and then on Ramp.
5. Adjust the Ion Focus value so that the 50/69 ratio is 0.8 or larger. The 219/69 ratio usually falls in the 60 to 80% range. When this PFTBA ion ratio is under 70%, the 174/95 ratio of BFB is usually under 100%.
6. In the Manual Tune window under the Calibrate menu item, adjust ion abundances.
7. Save the tune file with a new name, assign it to the method and verify that the tune passes by injecting a BFB sample according to the method requirements.

Conclusions

There are several ways to tune the Agilent 6890/5973 GC/MSD system to meet any of the USEPA BFB tuning criteria. However, factors such as source cleanliness, choice of column, flow rates and instrument-to-instrument variability make each GC/MSD system unique. Automated BFB and target tuning procedures are normally successful but the 174/95-ion ratio may not be high enough to meet laboratory needs. In our experience, the most robust and long-lasting BFB tunes were generated by the procedure outlined above under Modified Autotune. The procedure takes just a few minutes to complete.

References

1. *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*, Method 524.2, revision 4.1, U.S. Environmental Protection Agency, Office of Research and Development, National Exposure Research Laboratory, Cincinnati, OH (1995).
2. *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, Method 8260B, revision 2 (1996).
3. *USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLM04.2*, USEPA Contract Laboratory Program, Office of Emergency and Remedial Response.

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www.agilent.com/chem

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November 6, 2001
5988-4373EN



Attachment

V



Pace Analytical Services, Inc
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920.469.2436
Fax: 920.469.8827

VOA Calibration Process

Epic Pro

Make Q-Batch

- Batching -> New Batch -> Queue = MSV
- Click Empty Batch icon on taskbar
- Highlight QC Rule -> F9 -> type MSV
- Select appropriate QC Rule (i.e. MSV water) – Select OK – F10 to save
- Record Q Batch #

Create Standards

- System -> Utility -> Clone Standard by Event
- Select Event (111 = MeOH soil curve, 115 = Water/LLsoil curve) Select OK
- Double Click on standard event
- Review Standard composed of – Find/Replace if necessary
- Update expiration date to 7 days from creation – F10 to Save
- Operations -> Standard Log -> Enter – Record Standard #'s

Chemstation

Create Chemstation methods

- Tune MS, Save Tune file as date (i.e. 072513.u)
- Update both DBFB and Curve method to use new tune file
- Save Curve method as date (i.e. W072513.m)

Set up Sequence

- Load pre-existing curve sequence if available
- Change old method to the new method & copy through all files (DBFB remains the same)
- Change Q-Batch# in BFB, Curve and ICV files

Start Analysis

- Run minimum of 2 BFB injections to ensure the tune is optimized
- Retune or adjust as needed, repeat 2 more BFB
- Analyze a 2 blanks to verify the system is clean and IS areas within range
- First IS, pentafluorobenzene should be between 300,000 – 550,000 area counts
- Raise or lower EM as necessary – You **Must** reanalyze BFB if voltage was adjusted
- Reanalyze blanks to ensure correct voltage and proceed w/ analysis of curve

Target

Create Method

- Rename existing method to new name matching Chemstation method (i.e. W072513.m)
Note: If other data in Directory was processed w/ old method a copy of that method must remain in directory as well.
- To avoid excessive file size, Audit trail in method should be reset at a minimum of annually
The **ONLY** time an audit trail may be reset is prior to calibrating the instrument.
Note: The Audit trail will remain intact in previous days folder.
Double click into method folder, highlight the .audit file and delete

Edit Method

- Security -> Method unlocked
- Global -> Calibration – click “update Curve Parameters” to averaged
- File -> Zero Calibration
- Compound -> Edit Compound -> Calibration
Review all analytes to ensure all necessary points are enabled
Are any 300 points dropped? If so, mark them enabled and make note of these to change the
“Max Compound Amount Limit” after the curve has been run.
- Reports -> Tabular -> “Print Custom Report” –click “Select Format”
On toolbar a “Select” icon will appear
Click on ManIntprepost.mac – click “Open”
Note: It is necessary to do this **Every** time a calibration is zeroed, even if the macro shows up
in this field as the link to the macro that was lost when the calibration was zeroed.
- Sample -> Default Sample
Change “Lab Prep Batch” field to the new Q-Batch #
Change “Client SDG” to be the instrument and date (i.e. 40MSV2-07252013)
- Sample -> Surrogate/ISTD Parameter
Confirm that the correct IS/SS standard # is entered in the “Surrogate Lot#” field
Example – 51970:1.163 The 51970 is the IS/SS number followed by a colon followed by the
volume added (this is a fixed amount unless change to the standard delivery has occurred.)
- File -> Save Method
- File -> Exit

Process and Review Curve Data

- If significant Column maintenance was performed, it may be beneficial to process the 20 or 50 point first
to update RT's as the larger concentrations will have better spectra to confirm correct identification
- Select Method to calibrate and process files
Compound Sublist should be “all.sub”
Sample Type change to Calib Sample
Cal Level change to appropriate level 1-7
Double check that the Q-Batch # in MiscInfo and Lab Prep Batch are correct and match
Double check that the Client SDG reflects the instrument and date

- Review Target Data
Review each analyte of all points for correct spectrum, RT and appropriate integration
All Manual Integration of all curve points and ICV need to have Review Codes added
After reviewing all points, review each analyte point 1->7 to ensure consistent RT, spectra and Integration (i.e. shoulders cropped or included, etc.)

Review Curve in Target Method

- Edit Method
- Edit Compound -> Calibration
- Review each analyte to ensure Initial Calibration %RSD are less than 15.0%.
Note analytes >15% and re-examine target data for proper integration
- Check that all CCC compounds are less than 30% RSD
CCC's are 11DCE, chloroform, 12DiChloropropane, toluene, ethylbenzene and vinyl chloride
Instrument maintenance must be performed to correct problem if any >30%
If %RSD >15% and <30% note %RSD to record later.
- Check that all minimum relative response factors (RRF) were met for the SPCC – Chloromethane, 11DCE, bromoform are 0.1 and 1122PCA, chlorobenzene are 0.3– if any %RSD >15 note RRF to record later.
- If %RSD > 15 – Drop Upper or lower point to achieve %RSD < 15
If the Report Limit (RL) for analyte is not the 1 point, can the 1 point be disabled
Can the 7 point be dropped (or 6 & 7 points) – **Will require lowering Max Amount
Note: ONLY upper or lower points can be dropped, **NEVER** an intermediate point!!
Must have minimum of 5 points for Averaged RF curve
After disabling appropriate points – Click “Update Calibration” button
- If %RSD still > 15 – Switch Curve fit to Linear Regression
Change curve fit to Linear
**CCC Compounds (11DCE, chloroform, 12dichloropropane, toluene, ethylbenzene, vinyl chloride) MUST still be <30% RSD.
Initial Calibration R² must be 0.990 or greater
Must have minimum of 5 points for Linear regression curve
b intercept should be as close to zero as possible
i.e. by dropping the 300 point does the intercept go from 0.1980442 -> 0.0681234
This will give less false positive hits but require linear range to be lowered to 200ug/L
- If R² is not > 0.990
Change curve fit to Quadratic
***Must have minimum of 6 points**
R² must be 0.990 or greater
Like Linear regression the 300 point can be dropped (or 1 point added if RL is 5ug/L) to achieve the intercept closest to zero, as long as 6 points remain and linear range is adjusted.
- If calibration for compound will not pass
The Instrument cannot be run for lists including these analytes
Document analytes as failing in Run logbook
Place Post-It-Note on Instrument Terminal to alert other analysts of failures

Update Linear Range

- After all analyte curve fits have been checked
Compound->Edit Compound->Report Parm
Adjust "Max Compound Amt Limits" to reflect highest point used (300->200 if 7th point was dropped)
- Sublists -> Update Sublists
Check the "Update Sublists QC Limits" box
Highlight first sublist and hit Enter button
Arrow down to the next sublist and hit Enter
Repeat for all Sublists
**If you fail to update all the sublists, detects above linear range will not be "a" flagged in target
Epic Pro uses the "a" flag to switch Condition Code from "OK" to "OR"

Lock Method

- Security -> Initial Calibration Locked
- Note: Do not select "Method Locked" – This would not allow the method to be used to process data

Verify Initial Calibration

- View -> Initial Calibration
- This generates a report with calibration data that will appear on the lower tool bar
- Print report and review
 - The Calibration File Names in the header match the **correct** files used in the curve
 - All Average Response Factors < 15% and at least 5 points were included
 - All Linear Regression > 0.990 and at least 5 points were included
 - All Quadratic > 0.990 and at least 6 points were included
 - Are all low points dropped below Report Limit for that analyte
 - Any high points dropped verify that the Max on Column was lowered and Record max amount on the report
 - No midpoints of curve are missing
 - All CCC compounds averaged – If not is the %RSD < 30% - Record actual RSD on report
 - All SPCC minimum RF factors met – If not averaged, switch to Averaged in method record the RF on the report and switch curve back to appropriate curve fit
- Manually check individual Response Factors (RF) for at least one analyte
 - Calculate the RF for each point in the curve of an Averaged curve fit using the following formula
 - $RF = (\text{Area of analyte} * \text{concentration of IS}) / (\text{Area of IS} * \text{concentration of analyte})$
- Save method and Exit

Re-quantify and Uploading Curve and ICV

- Select Method
- Highlight Curve and re-quantitate
- Process ICV – Must use all.sub (or Full.sub)

- Review ICV and check CLP.rp
 - All SPCC Minimum RF must be met (if analyte is linear, must hand calculate)
 - All CCC Analytes must be <20%
 - All other analytes must be <30%
 - Note: Up to 5% (5 Analytes for a full list spike) may be between 30-40%)
 - All Analytes > 40% will be flagged as failing
 - Document analytes as failing in Run logbook
 - Place Post-It-Note on Instrument Terminal to alert other analysts of failures
- Generate all files to paperless (BFB, Curve and ICV)
- Upload all files to Epic Pro (double check Q-Batch is correct prior to upload)
- Check Q-Batch in Epic to ensure Curve, BFB and ICV imported correctly (may take several minutes)

MN Low Standard Verification

- Copy 1ppb, 5ppb & 20ppb files into another folder (i.e. the unprocessed blank following 300ppb)
- Paste all 3 files then Rename example (07251305.D -> MN01-07251305.D)
 - This will allow original files to be un-manipulated
- Cut files and paste back in original folder
- Re-Quant new MN files as LCS
 - Sample Type = QC Control Sample
 - Click QC SampleType
 - Sample Type = LCS
 - Spike List = MNLOW1.spk, MNLOW5.spk, MNLOW20.spk
- Highlight all 3 files and Do Quick Forms – Form 3 of LCS
- Print Form 3's and pass on to Supervisor to update MN report limits in Epic Pro

Before proceeding with analysis of samples

- Check Chemstation sequence that correct Q-Batch is in BFB and CCC
- Check that correct Method is referenced in the sequence

Attachment VI



VOA Calibration Review Checklist

Pace Analytical Services, Inc
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469 2436
Fax: 920 469 8827

Method:	SW846 8260B
Instrument:	
Q-Batch:	
HBN:	

Comments: Check box if there is an issue and document what was done.

Prior to Running Curve	Comments:
<input type="checkbox"/> IS/SS filled with Fresh standard and primed	
<input type="checkbox"/> Sparge tube/sipper tube clean or replaced	
<input type="checkbox"/> Replace Injection Port Septa if necessary	
<input type="checkbox"/> Instrument was tuned and new unique Tune file was created (i.e. 072513.u)	
<input type="checkbox"/> DBFB method was updated to use current tune file	
<input type="checkbox"/> Water method was updated to use current tune file and saved as the date (i.e. W072513.m)	
<input type="checkbox"/> Correct Q-Batch entered in sequence	
<input type="checkbox"/> Correct standards entered in sequence	
<input type="checkbox"/> Correct Method entered in sequence	

Prior to Processing Curve in Target	Comments:
<input type="checkbox"/> Method renamed to match Chemstation Method name	
<input type="checkbox"/> Method Unlocked and Zeroed	
<input type="checkbox"/> All point enabled (if 300 point was previously dropped)	
<input type="checkbox"/> All compounds curve fit re-set to Averaged	
<input type="checkbox"/> Manual Integration Macro selected	
<input type="checkbox"/> Q-Batch entered in Method and BFB default sample in the Lab Prep Batch field	

Prior to Processing Samples	Comments:
<input type="checkbox"/> Initial Calibration Report reviewed by another Analyst	
<input type="checkbox"/> Curve and ICV Passed - all failures recorded in logbook and note placed on instrument terminal	
<input type="checkbox"/> Sublist all updated to reflect linear ranges if 7th points were added or dropped	
<input type="checkbox"/> Files uploaded and generated to paperless	
<input type="checkbox"/> Q-Batch reviewed in EpicPro to assure properly imported	
<input type="checkbox"/> MIN Low Standard re-quanted and Quick Forms generated	
<input type="checkbox"/> Correct Q-Batch and Method are in new sequence	

Issues: Write any and all out of control issues below:

<input type="checkbox"/> Labtrack was issued	
<input type="checkbox"/> Supervisor was notified	

To the best of my knowledge, all of the above information is correct and all supporting documentation has been provided.

Analyst: _____ Date: _____
Reviewer: _____ Date: _____



Attachment 2-2
STAT Analysis Corporation

STANDARD OPERATING PROCEDURE 4010

**VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR BY /GAS
CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) (EPA Method TO-
14A/TO-15)**

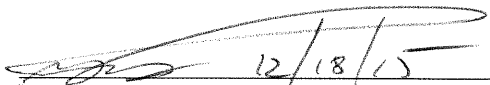
Revision 4

Effective Date: January 15, 2016


Printed Name

Signature/Date

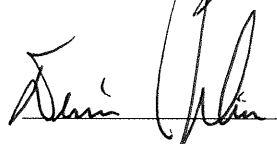
Vytautas Prasauskas
VOA – Air Supervisor

 12/18/15

Lory B Littlefield
QA Director

 12/18/15

Dennis Jachim
Technical Manager

 12/31/15

Bruce Gallant
Lab Director

 12/22/2015

**This Standard Operating Procedure has been prepared for the sole use of
STAT Analysis Corporation.**

Copy Number: _____

The absence of a Copy Number indicates this is an uncontrolled copy of the document supplied for information only.

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1.0 IDENTIFICATION OF TEST METHOD

SOP Title: Volatile Organic Compounds in Ambient Air by 2-Stage Thermal Desorption/Gas Chromatography/Mass Spectrometry (GC/MS) (EPA Method TO-14A/TO-15) is abbreviated as TO-14A/TO-15 in laboratory records.

2.0 APPLICABLE MATRICES

This method is applicable to air samples (source monitoring, ambient outdoor/indoor, etc.) in stainless steel or Silcosteel™ sampling canisters at sub-ambient (passively sampled) or positive pressure (actively sampled), Tedlar bags (or equivalent), or glass jars. If Tedlar™ bags are submitted and cannot be analyzed within 24 hours of sampling (for sulfur and chemically active compounds) or 72 hours (for chlorinated and aromatic compounds), the sample is quantitatively transferred to a canister for subsequent analysis.

3.0 DETECTION LIMITS

The laboratory SOP 1210 outlines the lab procedures for the determination of the MDL, LOD and LOQ. The MDLs determined by the lab and all supporting documentation are on file in the laboratory QA office.

Attachment 2 contains the current MDLs and reporting limits (RLs) for this SOP for TO-14A compounds and TO-15 compounds.

4.0 SCOPE AND APPLICATION

- 4.1 This SOP describes in detail the procedures used at STAT Analysis Corporation for the analysis of samples for Volatile Organic Compounds (VOCs) in air by GC-MS. This SOP as written is to be applied when TO-14A or TO-15 analysis is requested. This SOP is used to determine the concentration of VOCs in all types of air matrices.
- 4.2 SOP 4010 can be used to quantify most VOCs that have both boiling points below 220°C and are either insoluble or very slightly soluble in water. The SOP is also limited to compounds that elute as sharp peaks from a GC non-polar capillary column. Such compounds include low-molecular weight hydrocarbons, low molecular weight halogenated hydrocarbons, and aromatics. See Attachment 3 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.
- 4.3 The routine reporting limits (RLs) for this SOP are shown in Attachment 2. RLs will be proportionately higher for samples that require dilution or reduced sample size to avoid saturation of the detector. For special projects the reporting limits can be lowered, but must be within the analyte calibration range and not lower than its MDL.
- 4.4 This method is restricted to use by or under the supervision of analysts experienced in the use of a Gas Chromatograph/Mass Spectrometer (GC/MS) and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 SUMMARY OF TEST METHOD

- 5.1 Prior to analysis, the samples are prepared for the GC/MS using the sample preparation technique outlined in Section 14.1. VOCs are introduced into the Gas Chromatograph (GC) via a Tekmar Autocan Autosampler. A known volume of sample (usually 500cc) is first trapped on an adsorbent trap. The trap is then back-flushed with an inert gas (He) while being ballistically heated to transfer the analytes of interest to a cryofocuser. The cryofocuser is cooled in order to concentrate the analytes of interest into a small injection volume. The cryofocuser is then heated rapidly to transfer the sample to the chromatographic column.
- 5.2 This method describes chromatographic conditions that will allow for the separation of the compounds and for their qualitative and quantitative analysis by GC/MS. Identification of target compounds is accomplished by comparing component retention times to those of authentic standards and by comparing their mass spectra with the spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a calibration curve containing a minimum of 5 points for each target compound.
- 5.3 **Method Modifications from Reference** – This SOP is based on EPA Methods TO-14A and TO-15 with the following modifications:
- 1) Calibration standards are prepared using a static dilution system. EPA Method TO-15 recommends a dynamic dilution system. Static dilution is used to minimize the cost of standards preparation. Static dilution has been demonstrated to be as accurate as dynamic dilution through the periodic testing of performance evaluation samples.
 - 2) The leak test procedure was modified to a canister pressure drop of < 1.0 psi over 12 hours, instead of < 2.0 psi over a 24-hr. period.
 - 3) All blanks, QC samples (LCS, LCSD) and samples must have internal standard responses within $\pm 40\%$ of the daily CCV internal standard responses. (Method states $\pm 40\%$ of the internal standard responses from the most recent valid calibration.) ICAL internal standard response for each calibration standard must be within $\pm 40\%$ of the mean internal standard response of the ICAL.
 - 4) EPA Method TO-14A specifies the use of a 3-point calibration curve with the addition of a humidified zero air reference point. We will be using a minimum of 5 points for the calibration curve; however, the zero point will not be included because we will be using internal standard (rather than external standard) calibration- see modification 5 (following).
 - 5) EPA Method TO-14A does not require the use of internal standards. The three internal standards specified by EPA Method TO-15 will be used.
 - 6) In cases where QA/QC criteria differ between EPA Methods TO-14A and TO-15, the more stringent QA/QC criteria will be employed.
 - 7) In cases where a compound is listed only on EPA Method TO-15, the quality control specifications from TO-15 will be applied to only those compounds.
 - 8) The clean canisters are evacuated to <0.05psia (2.6mm Hg) the method specifies <0.001psia (0.05mm Hg).

6.0 DEFINITIONS

The STAT Analysis Quality Manual Section 19.0 contains all of the definitions of standard terms used in SOPs.

7.0 INTERFERENCES

- 7.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Interferences will vary from source to source depending upon the particular sample being tested. Determine if the source of interference is laboratory induced. If the source of the interference is laboratory induced, take corrective action to eliminate the problem.

- 7.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. A Method blank (zero air or nitrogen) must be analyzed to demonstrate that carryover has been eliminated. Reanalysis must be performed on all affected samples to demonstrate that the compound concentration is from the sample and not due to carryover. The sampling system may require extensive bake out and cleaning after a high-level sample. Cleaning may include trap replacement, purge gas filter replacement, and/or transfer line cleaning/replacement.
- 7.3 The sampling system can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sampling system. The system is leak-tested daily to ensure that compounds potentially present in the laboratory environment do not contaminate the system. A blank is prepared in a previously cleaned and certified sampling canister. It is analyzed daily to ensure that system is not contaminated. Because of the design of air sampling canisters, it is extremely rare for canisters to become contaminated during shipment/transport to the lab.
- 7.4 All sampling canisters are cleaned in batches on a manifold. They are cleaned using repeated cycles of pressurization with humidified zero air or nitrogen followed by evacuation. All canisters in the batch are pressurized to 30psia (15.3 psig) with humidified zero air or nitrogen and the canister valves are closed. The canisters are allowed to stand overnight, and then the pressure in each canister is individually checked. A pressure-drop of >1.0psi in any canister over a 12-hour period is indicative of a leak. Any leaking canister should be taken out of service and tagged for a leak test. If the canister can be repaired and subsequently demonstrated to pass the leak test, it can again be cleaned and certified and placed back in service. At least one canister per batch is certified by final pressurization to 30psia (15.3 psig) with humidified zero air or nitrogen, followed by analysis on the GC/MS system. The canister must contain less than the reporting limit of each target compound in order to be considered clean. Cleaning cycles are repeated for ALL canisters in the batch if the selected canister fails certification criteria. Cleaned canisters can be stored under pressure for unlimited time and evacuated to -14.65 psig as needed prior to sampling. Evacuated canisters stored in the laboratory can be sent to collect field samples within 30 days after evacuation, after that time, unused evacuated canisters must be checked and reevacuated as necessary.
- 7.5 Any apparatus used for sample collection must be cleaned between every use.
- 7.6 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free of contamination under the conditions of analysis by running calibration and reagent blanks. The use of non-PTFE plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided. Pressure regulators with stainless steel diaphragms should be used for all cylinder gasses. Carrier gas for the GC/MS system should be passed through moisture, hydrocarbon, and oxygen traps.

8.0 SAFETY

The concentrations of hazardous chemicals in most air samples are minimal and the analyst should not be exposed to the sample due to the closed nature of the sample canisters and analytical system; however, numerous compounds analyzed by this method are known or suspected carcinogens. Proper personal protective equipment including safety glasses and a lab coat are required. Other safety precautions must be conducted in accordance with the SAP 003 Chemical Hygiene Plan. Other actions can also be applied if deemed necessary. A reference file of Safety Data Sheets (SDS) is available to all personnel involved in this method. This method requires the safe handling and use of pressurized cylinders.

9.0 EQUIPMENT AND SUPPLIES

The following apparatus is recommended for performing this procedure. Equivalent items can be used, if with their use, the analytical and QA/QC requirements in this SOP can be met. All catalog numbers in this SOP are current as of the effective date and thereafter subject to change.

9.1 Gas Chromatograph/Mass Spectrometer system (GC/MS)

- 9.1.1 Gas chromatograph(GC) – Agilent 6890 electronic flow control gas chromatograph with the column directly coupled from the transfer line to the source or equivalent
- 9.1.2 Column – 60 m X 0.320 mm ID Rtx-1 column with 1 µm film thickness or equivalent.
- 9.1.3 Mass spectrometer(MS) - Agilent 5973N or equivalent.
- 9.1.4 Data system – A computer system using Agilent 3365 Chemstation Software (G1701AA Version C.00.00 or more current) is used to collect and process data or equivalent.
- 9.1.5 Tekmar #64 Air Toxics trap or equivalent.
- 9.1.6 Tekmar #14-6765-003 Air Toxics trap or equivalent.
- 9.1.7 NIST Library of Reference Spectra.
- 9.2 Tekmar Autocan canister autosampler or equivalent.
- 9.3 Microliter syringes - 10 µL, 100µL and 1000 µL.
- 9.4 Sample canisters - Restek TO-Can or equivalent.
- 9.5 Cleaning manifold – in-house design capable of automated cleaning of canisters in batches of at least 8.
- 9.6 Pressure gauge capable of measuring at 0.05 psia (2.6mm Hg) within 20%.
- 9.7 Pressure gauge ± 1% accuracy, -14.65 psig (30" Hg)/ 0-30 psi.
- 9.8 Air sampling apparatus – passive air sampling kits (e.g., Restek passive air sampling kit).
- 9.9 Gas Supplies
 - 9.9.1 Ultra-High Purity Helium (99.999%)
 - 9.9.2 Reagent Grade Zero air or ultra grade air or nitrogen
 - 9.9.3 Liquid Nitrogen
 - 9.9.4 Hydrogen (optional carrier gas replacement for helium).
- 9.10 Hydrocarbon filters, Agilent model HT-200-2 or equivalent

10.0 REAGENTS AND STANDARDS

The following reagents and standards are required to perform this procedure. When instructions are given on how to prepare a specific volume of a reagent or standard, larger or smaller volumes can be prepared as needed so long as the final concentrations remain the same—this is especially applicable if standards are being prepared in 1L canisters as opposed to the usual 6L canisters. Any other deviations from the reagents or standards listed in this SOP could be detrimental to the quality of the data produced. Such deviations would have to be approved and documented (see 230 Corrective Action SOP). Instructions for labeling and record keeping of reagents and standards are contained in SOP 1010 Analytical Standards and Reagents Receipt and Preparation.

NOTE: All concentration listed in Table 1 are nominal concentrations. Any stock standard where an individual compound differs from the listed nominal concentration by greater than 10% will be rejected or the spike value in LIMS must be corrected to match the reported concentration on the COA.

NOTE: Zero Grade air or ultra grade air or nitrogen must be logged in and tracked as a reagent

- 10.1 Analytical reagent grade chemicals are used in all tests. Unless otherwise indicated, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
- 10.2 Organic-free reagent water - All references to water in this SOP refers to Type II reagent water (in-house system). Note: Reagent water may be de-gassed with High Purity Helium to remove trace impurities.
- 10.3 Stock Standard mixtures - Standard mixtures are purchased in cylinders from certified vendors traceable to NIST, where possible. The reagents and standards must meet SOP 1330 section 14.1.2. Calibration standards and Quality Control Standards (see section 10.4) are prepared directly from the Stock Standard mixtures in clean, certified 6L canisters, diluted to appropriate concentration with zero grade air or nitrogen, and humidified with reagent water. The prepared standards must be replaced monthly or sooner if comparison with quality control check samples indicates a problem. Quality Control Standards must be from different source (lot numbers) than Calibration Standards.
- 10.3.1 The primary stock solution BFB is available in cylinders with internal standards at 100ppbv and in vials containing 5000µg/ml of BFB in methanol. See section 10.6 for preparation.
- 10.3.2 The internal standard mix contains 100 ppbv each of bromochloromethane, 1,4-difluorobenzene, and Chlorobenzene-d5 and can be combined with 100ppbv BFB.
- 10.4 Calibration Standards** – A minimum of five calibration standards are required. Canisters containing calibration standards are prepared at concentrations suitable to provide necessary trapping volumes for the following eight levels of calibration standard concentrations: 0.2ppbv, 0.5ppbv, 1.0ppbv, 2.0ppbv, 5.0ppbv, 10ppbv, 25ppbv and 50ppbv for routine RLs or lower concentrations for special projects. (See example in Table 1).
- 10.4.1 A static dilution system is used to prepare the calibration standards. The system incorporates a very accurate (0.1% gauge) test pressure gauge and uses pressure differential to calculate dilutions. This method of standard preparation is documented to be equivalent to dynamic dilution.
- 10.4.2 Working standards are prepared by pressurizing a clean, certified, evacuated canister to just above 0psi on the test pressure gauge. The pressure differential on the gauge for standard preparation is 1.5psi for the 5ppbv calibration standard, and 15psi for the 50ppbv calibration standard. Finally, the canister is pressurized to 30psia (15.3 psig) final pressure using zero grade air or nitrogen. The trapping volume required for each calibration level is given in Table 1.
- 10.4.3 Calibration standards and quality control standards in canisters can be used for one month from the date of preparation. The pressure in the canister should be checked periodically in order to prevent the system from appearing to be out of control due to low standard pressure. The 2, 5 and 10ppbv level standards are rotated on a subsequent day for CCVs.

Table 1 Calibration Standards

Desired conc. (ppbv)	Trapping volume of standard mix	Trapping volume of ISTD	Calibration Canister Used
50	500	50	50ppbv
25	250	50	50ppbv
10	100	50	50ppbv
5	250	50	10ppbv
2	100	50	10ppbv
1	50	50	10ppbv
0.5	250	50	1ppbv
0.2	100	50	1ppbv

- 10.5 Internal Standards: The internal standards used are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d₅. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Attachment 3, e.g. for 1,4-difluorobenzene, use 114 m/z for quantitation). If interferences are noted, use the next most intense ion as the quantitation ion.

- 10.6 GC/MS tuning standard – BFB comes in the cylinders together with internal standards at 100ppbv concentration. 50cc of this mixture is analyzed separately or together with CCV standard to check instruments tune. Using the Primary stock solution BFB (5000 µg/mL in methanol) a 6-L canister is prepared containing enough BFB to quantitatively transfer 50ng or less of BFB in a known volume to the analytical system. The BFB-containing canister is prepared by injecting 1.0µl of the stock BFB solution into a 6-liter canister and pressurizing the canister to 30 psia with zero air or nitrogen. Each 90cc sample from this canister contains 36ng of BFB. At this concentration, BFB is very stable in the canister and can be used up to 6 months. The absolute pressure in the BFB canister should be checked each time it is used. When the pressure falls below 0 psig in the canister, the response of BFB will start to drop and the spectrum may not pass tune requirements. Prepare a new canister of BFB when the pressure in the current canister falls to 0 psig. Failure to check the pressure in the BFB canister could lead to unnecessary instrument maintenance and downtime. Alternatively, performance of BFB can be demonstrated within the CCV standard; for 2ppbv and 5ppbv CCV standard add 0.2µl of BFB standard solution, for 50ppbv CCV standard add 0.9µl.
- 10.7 The Laboratory Control Sample/Laboratory Control Sample Duplicates (LCS/LCSD): An LCS/LCSD must be analyzed with every sample batch. Spiking solutions are prepared from certified analytical stock standard solutions purchased independently from the Calibration Standards, if purchased from the same supplier must have different lot number. The LCS Spiking mixtures are prepared in the same manner as the calibration mixtures (See section 10.4 and Table 1). The LCS is also used for initial calibration verification (ICV). The concentration of the LCS and LCSD is 5ppbv.
- 10.8 Method Blank. A canister containing zero air or nitrogen will be analyzed during each series of analytical runs. Each sequence will have an evaluation of the BFB tuning solution, together or followed by a continuing calibration check, and then the blank canister. The blank must contain less than the reporting limit of each target analyte for analysis to continue.
- 10.9 Reagent Grade Zero Air or nitrogen.

11.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 11.1 Containers used to collect samples for the determination of volatile organic compounds: clean, certified air sampling canisters, Tedlar bags, glass jars, adsorbent tubes. Adsorbent tube analysis will not be covered in this SOP. Tedlar bags, glass jars and air sampling canisters are available from a variety of vendors.
- 11.2 All samples are generally kept free of contamination because the sample containers must be leak-free. See section 14.9 for details of how to perform the leak-test procedure. As a precaution, samples should be stored away from standards, reagents, or other laboratory chemicals that could cause cross-contamination.
- 11.3 Sample analysis must be within 30 days from the date of sample collection. After client approval or 30 days have elapsed, the sample will be disposed.

12.0 QUALITY CONTROL

The following details the QC requirements that apply to this analysis. Each Quality Control Indicator (QCI) provides information pertaining to either method or individual sample performance. Our goal is to produce defensible data of known and documented quality. The results of these QCI samples are used to assess the acceptability of data.

12.1 Calibration Verification

An Initial Calibration Verification (ICV) standard containing all of the target compounds reported in this method (refer to section 10.7) is analyzed immediately after the completion of the initial calibration. The ICV is purchased from a second source to verify compound concentrations. Continuing calibration verification is performed daily by rotating the 2ppbv, 5 ppbv, and 10 ppbv .primary calibration standards.

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12.2 Blanks

Method Blank analysis is performed to determine if any contamination is present in the analytical process and is used to evaluate acceptance of the batch of samples. A method blank is analyzed once per 24-hour period per matrix type. The method blank is processed through all preparatory steps used for the samples. The blank is analyzed using the same instrument and conditions as the samples.

12.3 Laboratory Control Sample (LCS)

The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The LCS is analyzed once per batch of samples not to exceed the 24-tuning clock. The LCS is analyzed using the same instrument and conditions as the samples. Refer to section 10.7 for LCS compounds and concentrations. All target compounds are evaluated in the LCS.

12.4 Surrogates

Not applicable to this method.

12.5 Duplicates

Duplicates of field samples or of the LCS must be prepared in compliance with the method requirements and client directives. Replicate samples will be analyzed whenever the client provides them. However, when they are not, then the LCS/LCSD pair must be analyzed per batch of 20 incoming samples. In both cases, reproducibility performance is defined as $\pm 25\%$ RPD.

Note: It is an option to analyze the same can as a duplicate. The same acceptance criteria would apply.

12.6 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSDs indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. This information does not determine the validity of the entire analytical batch. Customers may request MS/MSDs be analyzed or periodically the laboratory can request that the customer collect additional samples in support of the laboratory's internal QC program. The MS/MSD pair shall be analyzed using the same instrument and conditions as the samples.

12.7 Internal Standards

The internal standards used for this method are added to all samples, standards, and blanks. Refer to section 10.5 for preparation and concentration instructions. The internal standard responses for all samples including QA/QC samples (blank, LCS, LCSD, etc.) must be within $\pm 40\%$ (60 to 140%) of the daily calibration check internal standard responses. If any sample is run in which the internal standard responses fall outside of these criteria, the sample will be re-analyzed. If, upon re-analysis, the internal standard response(s) still fall outside of these criteria, the standard will be re-prepared if possible (blank, LCS, or LCSD) or if the criteria are not met for a client submitted sample, a note will be made in the case narrative of unacceptable internal standard response(s).

13.0 CALIBRATION AND STANDARDIZATION**13.1 Mass Spectrometer Tuning Verification**

The Mass Spectrometer is software-tuned with the BFB tuning macro using perfluorotributylamine (PFTBA). Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for a 50 ng or less injection of BFB. Analyses must not begin until all these criteria are met. From the ISTD/BFB canister 50cc of standard are trapped that contains 36ng BFB, or from BFB canister, 90cc are trapped that contains 36ng BFB. Analyze the solution using the GC/MS instrument conditions for sample analysis shown in section 14.2.1.

Background subtraction must be straightforward and designed only to eliminate column bleed or instrument background ions. Obtain a mass spectral analysis, using the method listed above, of BFB and confirm that all the

key m/z criteria in Table 2 are achieved. The mass spectra of BFB are acquired using the following methods: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged, the peak apex only is evaluated, or the entire BFB peak is averaged. If background subtraction is required, this must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Note: the instrument-tuning file used to acquire the BFB criteria must be also used to acquire QC and sample data. If the criteria are not passed, the analyst must perform needed maintenance, retune the mass spectrometer and repeat the test until all criteria are achieved.

Note 1: The 24-hour tune period begins at the start of the BFB analysis, which is recorded as the data acquisition start time on the data file. The last valid calibration standard in the ICAL must have a data acquisition start time less than 24 hours from the start of the tune period.

Table 2 Acceptance Criteria for BFB

Target Mass	Relative to Mass	Lower Limit%	Upper Limit%
50	95	8	40
75	95	30	66
95	95	100	100
96	95	5	9
173	174	0	< 2
174	95	50	120
175	174	5	9
176	174	93	101
177	176	5	9

13.2 Initial Calibration (ICAL)

- 13.2.1 Analyze each calibration standard according to the procedure listed in Section 14 (standards are analyzed under the same conditions as samples) and tabulate the target ion area of the compound against concentration for each compound relative to the appropriate relative internal standard. See Attachment 4 for the list of internal standard assigned to each compound. Calculate response factors (RFs) for each compound in each calibration standard as follows:

$$RF = \frac{A_x * C_{is}}{A_{is} * C_x}$$

where:

- A_x = Area of the characteristic ion for the compound being measured.
 A_{is} = Area of the characteristic ion for the specific internal standard.
 C_{is} = Concentration of the specific internal standard (10ppbv for this method)
 C_x = Concentration of the compound being measured (ppbv).

- 13.2.2 Calculate the average response factor (\overline{RF}) for each compound as follows:

$$\overline{RF} = (\sum RF_i \text{ to } RF_n) / n$$

where:

- RF = RF for each of the calibration levels (minimum 5)
 n = Number of RF values (minimum 5)

- 13.2.3 Calibration Check Compound (CCC) responses are monitored to check the integrity of the system. All compounds are considered calibration check compounds.

Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for the CCCs.

To calculate %RSD: $\%RSD = 100\% \times SD/\overline{RF}$

where:

%RSD = % Relative Standard Deviation

\overline{RF} = average of initial RFs for a compound

SD = standard deviation (n-1) of average RFs for a compound

To calculate Standard Deviation (n-1 degrees of freedom)

$$SD = \sqrt{\sum_{i=1}^N \frac{(RF_i - \overline{RF})^2}{N-1}}$$

where: RF_i = RF for each of the calibration levels

N = Number of RF values (minimum 5)

- 13.2.4 The %RSD of all CCC compounds should be < 30%. If the response of any CCC compound varies by more than 30%, the system is considered too reactive for analysis to continue. Two exceptions of up to 40% are permitted. This exception will be applied only to analysis by EPA Method TO-15. Perform the initial calibration procedure beginning with section 13.2.1.
- 13.2.5 Linearity : If the %RSD of any compound is 30% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor is used for quantitation.
- 13.2.6 Retention Times: The Relative Retention Times (RRT) of each compound in each calibration standard should be within ± 0.06 RT units.
- 13.2.7 Internal Standards: For the Initial Calibration, the response for each internal standard must be within $\pm 40\%$ of the mean internal standard response for all calibration standards.
- 13.2.8 Before sample analysis can begin, a Initial Calibration Verification (ICV) (see Sec. 10.7) must be performed. The ICV results must be within the acceptance criteria as established for the CCV compounds (Section 13.3).
- 13.2.9 NOTES:
1. All calibration curves must have a unique identifier. The calibration date is incorporated into the method identifier for easy reference.
 2. All initial calibration standards must be analyzed within the 24-hour BFB tune verification window.
 3. Calibration must be completed (all standard levels analyzed) within 24 hours or the entire calibration sequence must be repeated. All initial calibration standards must be analyzed and verified prior to field sample analyses.
 4. Do not add calibration standards and update the curve after the original calibration date.
 5. Multiple analyses of the same calibration level are not allowed to be included in the calibration curve.
 6. Compound responses cannot be selected from different analyses of the same calibration level (i.e. all compound responses for level 1 must come from the same data file). If more than five calibration levels are analyzed, it is permissible to either drop the low compound concentration (poor response) or the high

- compound concentration (detector saturation). Do not drop a point in the middle of the curve except as in Item 7.
7. If more than five calibration levels are analyzed, it is permissible to drop an entire level if the analyst can document a specific reason or reasons why that level should be excluded. Mechanical failures, significant loss of all compound responses possibly due to poor technique in standard preparation, or software malfunction during data acquisition (system did not acquire all compound data) are plausible reasons. The reason must be justified and documented. The supervisor should review this situation and approve the analyst's decision.
 8. The need to reanalyze the same calibration level more than once indicates a potential problem with the Working Standard mixture. Prepare a fresh Working Standard and repeat all calibration levels. All calibration standard levels must come from the same Working Standard.
 9. Manual integration must only be used if the software fails to properly integrate the peak (See SOP 1255 Manual Integration). If manual integration is used for a particular compound, all levels of that compound do not have to be manually integrated. Example: the computer generated integration of a compound in the low level standard is not technically sound. A manual integration of the compound is performed (due to baseline noise, etc) in order to achieve a technically sound integration. The computer integrates other levels of the compound correctly. The manual integration of the low level must match, as best as possible, the computer generated integrations for all other levels.
 10. If any compound in the mid-point level is manually integrated, the integration of this compound must be reviewed for every continuing calibration standard and QC check solution (e.g. LCS) to ensure that the integration is the same as the manual integration performed in the initial calibration (ICAL) mid-point. A consistent manner of integration must be achieved.
 11. All manual integrations must be reviewed and approved according to SOP 1255 Manual Integration.

13.3 Continuing Calibration Verification (CCV)

- 13.3.1 The mass spec must pass tune criteria (see Section 13.1) every 24 hours during analysis of samples.
- 13.3.2 A continuing calibration verification (CCV) sample is analyzed every 24 hours during analysis of samples. Different standards are analyzed for the CCV such that a wider range of the calibration curve is verified. For example: on successive days analyze the 2ppbv, the next day the 5ppbv standard solution is analyzed, and the following day, the 10ppbv standard solution is verified. Compare the response factors and the CCCs, and the internal standards from the CCV with the following criteria.
- 13.3.3 Control limits for the CCV compounds are $\pm 30\%$ difference from the expected concentrations.
- 13.3.4 The areas of the internal standards in the CCV must be between 60% - 140% of the respective areas of the internal standards in the corresponding level in the ICAL.
- 13.3.5 The retention times of the internal standards in the CCV must be within ± 20 seconds of the respective retention times of the internal standards in the ICAL mid-point calibration standard.
- 13.3.6 If the CCV results obtained are outside the acceptance criteria, corrective actions must be performed. If routine corrective actions fail to produce an acceptable *second consecutive (immediate)* CCV, then either the lab has to demonstrate performance after corrective action with two consecutive successful CCVs, or a new ICAL must be performed. If the instrument has not demonstrated acceptable performance, sample analyses cannot continue until a new ICAL is established and verified with an ICV. However, sample data associated with an unacceptable CCV may be reported as qualified data under the following special conditions:
 - 13.3.6.1 When the acceptance criteria for CCV are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification must be reanalyzed after a new ICAL has been established, evaluated and accepted.
 - 13.3.6.2 When the acceptance criteria for the CCV are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples

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affected by the unacceptable verification must be reanalyzed after a new ICAL is established and verified with an ICV.

13.3.6.3 When the acceptance criteria for the CCV are exceeded and it is not possible to reanalyze the sample due to limited sample quantity and the laboratory cannot obtain a new sample, the data may be reported with the appropriate data qualifiers if the client has been contacted and agrees to accept the qualified data.

13.4 Records: Initial and Continuing Calibration Records will contain, at a minimum, the following:

- 1) Calibration date
- 2) Test method
- 3) Instrument
- 4) Analysis date
- 5) Each compound name
- 6) Analyst's initials or signature
- 7) Standard Concentration (appropriate units) and number of standards
- 8) Response (appropriate units)
- 9) Calibration curve or response factor
- 10) Evaluation of and Statistics for ICAL curve fit in order to judge calibration curve acceptance
- 11) Evaluation of and Acceptance Limits for ICV analysis in order to judge calibration curve acceptance.
- 12) Evaluation of and Acceptance Limits for CCV analysis in order to judge continuing calibration acceptance.

14.0 PROCEDURE

14.1 Sample Preparation

14.1.1 Incoming samples will have their pressure checked using the test pressure gauge. Samples collected using a passive sampler should have a pressure higher than -7.35 psig. Samples collected using an active (pump equipped) sampler should have a pressure lower than 40 psig. Samples with pressure of -7.35 psig or less should be accepted and client should be contacted to find if sample analysis should be processed.

If Tedlar bag is submitted, it can be directly attached to the instrument or, if the analysis cannot be performed within the Tedlar bag hold time (depending on compounds of interest 24 or 72 hours) then the contents will be transferred to a clean, evacuated canister.

14.1.2 Zero air or nitrogen can be added to each incoming canister sample, the initial and final pressure of every sample is recorded. The initial and final sample volumes are recorded in LIMS and used to calculate the sample prep factor. The level of pressurization will affect detection limits; and depends on project objectives. The following formulas are used to calculate prep factor:

$$V_S = \frac{V_C \times P_S}{14.7} ; \quad V_F = \frac{V_C \times P_F}{14.7} ; \quad PF = \frac{V_F}{V_S}$$

where:

PF - prep factor,

P_S - pressure of incoming canister, psia,

P_F - final pressure of the canister after preparation, psia,

V_S - sample volume in the canister, ml,

V_F - final gas volume in the canister after preparation, ml,

V_C - volume of the canister, ml

14.1.3 If the sample pressure in the canister is too low to meet needed reporting limits the client will be contacted.

14.2 GC/MS Analysis

14.2.1 GC/MS Conditions:

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GC/MS operating conditions	
EMV	1100-3000
Mass Range	35-300
Scan Sampling Rate	2 [^] 2
Initial Column Temp.	25°C
Initial Temp Holding Time	3 minutes
Ramp 1 Temp Program	10°C/minute
Ramp 1 Final Column Temp.	230°C
Ramp 1 Holding Time	2 minutes
Ramp 2 Temp Program	-
Ramp 2 Final Column Temp	-
Ramp 2 Holding Time	0 minutes
Ramp 3 Temp Program	-
Ramp 3 Final Column Temp	-
Ramp 3 Holding Time	-
Injector Temp.	n/a
Source Temp.	230°C
GC/MS Interface Temp.	230°C
Split Ratio	n/a
Carrier Gas	UHP Helium

Note: Blanks and LCS/LCSDs will have trapping volumes of 500 cc. Undiluted samples will have trapping volumes of 500 cc. For projects that require very low detection limits, a greater trapping volume may be used. The same volume is trapped for the LCS, LCSD and method blank. The volume trapped of diluted samples will be noted.

14.2.2 Tekmar Autocan operating conditions:

GC Start Option	End of Desorb
GC Cycle Time	19.5 minutes
Cryo	On
Line Temp	150 °C
Valve Temp	150 °C
MCS Line Temp	40 °C
Trap Standby Temp	150 °C
Cryo Standby Temp	130 °C
MFC Standby Flow	20
Trap Cool Temp	30 °C for 6" trap; 35 °C for 12" trap
MFC Transfer Flow	50
Drypurge Time	2 minutes
Drypurge Temp	40 °C
Drypurge Flow	100
Desorb Preheat Temp	30 °C for 6" trap; 35 °C for 12" trap
Trap Desorb Time	4 minutes for 6" trap; 3 minutes for 12" trap
Trap Desorb Temp	275 °C for 6" trap; 245 °C for 12" trap
Cryo Cool Temp	-178 °C
Cryo Inject Time	1 minute
Cryo Inject Temp	130 °C
Trap Bake Time	10 minutes
Trap Bake Temp	310 °C for 6" trap; 280 °C for 12" trap
MCS Bake Temp	60 °C

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MCS Cool

40 °C

- 14.2.3 Perform Standard Spectra Auto-tune with the GC oven at 100 °C to check for air and water leaks in the GC/MS system.
- 14.2.4 Access the Teklink software to generate a sample analysis sequence.
- 14.2.5 Access the Chemstation software to generate a sample analysis sequence. (This must match the Teklink sequence.)
- 14.2.6 Analyze the BFB tuning standard and verify that the tuning requirements from section 13.1 Table 2 are met.
- 14.2.7 Analyze the Continuing Calibration Verification Standard (CCV) and evaluate using CCV acceptance criteria (See Section 13.3). If target parameters achieve these limits, recovery and retention times of the compounds are within acceptance limits, and the chromatography (baseline noise and compound tailing) is acceptable then continue the analysis. If any one of these criteria is not met, take appropriate corrective action to identify and correct the problem
- 14.2.8 Analyze the Method Blank. The method blank must contain less than the reporting level for all target compounds. In addition the response of the internal standards must be within $\pm 40\%$ of the internal standard response of the daily calibration check (CCV).
- 14.2.9 Analyze the LCS (and its duplicate, if applicable). The percent recovery must meet the criteria listed in section 18.2. If the LCSD is analyzed, it must meet the %RPD criteria listed in section 18.4.
- 14.2.10 If all criteria are met, continue with sample analysis.
- 14.2.11 Analyze the sample replicates, when provided by the client. The RPD must meet criteria specified in section 18.4.
- 14.2.12 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, sample dilution and reanalysis is an option. Variable trapping volume on the Autocan can be used to accomplish sample dilutions of up to 1:25. Trapping 50cc of sample (rather than 500) while still trapping 50cc of the internal standard mixture will yield a 1:10 dilution.
- 14.2.13 Perform all qualitative and quantitative measurements as described in Sections 14.4 and 14.5. Samples may be retained for up to 30 days from the date of sample collection for repeat analysis should the sample fail data review for any reason. After client approval or 30 days has elapsed, the canister should be cleaned along with a batch of other canisters, and one canister from each batch should be certified as described in section 7.4.

14.3 Analytical Sequence

START CHEMSTATION SOFTWARE:

From Desktop: Start/Programs/MSD ChemStation/Instrument #1/VOA5 (or VOA6)

The software will locate the instrument and all peripheral equipment. Once the software is fully loaded there will be two windows visible. Window one is the instrument control window. Instrument parameters are readily viewed from this window. The second window is the sequence control window. No instrument parameters are viewable from this window.

SEQUENCE SETUP

From menu: Method/Load

1. Load the method you want to use for analysis (e.g., A112102.m)
2. OK (to save changes)

From menu: Sequence/Run

3. Set current date in Data File Directory space (i.e., C:\MSDCHEM\2\DATA\112402\)
4. OK (to save changes)

From menu: Sequence/Edit Sample Log Table

1. Set data file name using current date using the following format MMDDYY##.d (e.g., 11240202.d)
2. Set up sequence order using the following format: (**NOTE:** Tune and CCV must always run first, followed by analysis of the method blank. The only exception to this is the ICAL sequence.)

LINE	TYPE	VIAL	DATA FILE	METHOD	SAMPLE NAME-INSTRUMENT
1	Sample	1	11240201	A112102	TUNE BFB112402-5
2	Sample	1	11240202	A112102	CCV CCV112402-5 5.0
3	Sample	2	11240203	A112102	MBLK MB112402-5
4	Sample	3	11240204	A112102	LCS LCS112402-5
5	Sample	4	11240205	A112102	LCSD LCSD112402-5
6	Sample	5	11240206	A112102	SAMP #####
7	Sample	6	11240207	A112102	SAMP #####
...	Sample	A112102	...
n	Sample	k	112402##	A112102	SAMP #####

NOTE:

a) The number of samples, including tune and CCV that can be analyzed within the 24-hour window varies with each analysis, but can not exceed 20 excluding their dilutions. Use this list as a guide but always check that the samples were analyzed within 24 hours of the BFB injection. The TUNE can be assessed from BFB in the CCV.

b) Analytical sequences containing only cleaning batch certification samples, require a TUNE and CCV, no LCS/LCSD required.

3. OK (to save changes)

From menu: Method/Set New Default Paths

1. Update the method and data file paths using the current date (i.e., C:\MSDCHEM\1\DATA\060702\)
2. OK (to save changes)

Using Windows Explorer copy all of the method files from the previous day's folder into the current day's folder.

Note: After each ICAL, the method is saved using the following format: matrix instrument MMDDYY of last ICAL (e.g., A112102.m).

TO START SEQUENCE:

From menu: Sequence/Run/Run Sequence

Start of the 24 hour sequence (as documented as the date/time stamp on the data file for the Tune Standard).

The analytical sequence for Initial Calibration is:

BFB (Tune Standard)

Calibration standard (S1) or Calibration standard (S1) and BFB

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Calibration standard (S2)
Calibration standard (S3)
Calibration standard (S4)
Calibration standard (S5)
Calibration standard (S6)
Calibration standard (S7)
Calibration Standard (S8)
Initial Calibration Verification Standard (ICV) (Can be used as LCS)
Method Blank (MBLK)
LCS Duplicate (2nd ICV, prepared in separate canister)
Samples #2, #3, etc. until the end of 24 hour tune period

Additional levels can be added for special project analysis.

The analytical sequence for Sample Analysis is:

BFB

Continuing Calibration Verification Standard (CCV) or CCV and BFB

Method Blank (MBLK)

LCS (ICV)

LCS Duplicate (2nd ICV, prepared in separate canister)

Sample #1

Samples #2, #3, etc. until the end of 24 hour tune period

14.4 Data Interpretation - Qualitative Analysis

- 14.4.1 A compound is identified based upon Relative Retention Time (RRT) and by comparison of the sample mass ions with the mass ions of a standard of the suspected compound (standard reference ions). The reference mass ion ratio must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass ion are defined to be the two or three ions of greatest relative intensity. See Attachment 3 for the ions to be monitored. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC relative retention time (RRT) as those of the standard component; and (2) correspondence of the sample component and the standard component mass ions. Intensities of the characteristic ions must maximize in the same scan or within one scan of each other. In cases where there is interference of the characteristic ions, alternate characteristic ions may be chosen, after they have been appropriately evaluated.
- 14.4.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.
- 14.4.3 The relative intensities of the characteristic ions must agree within specified range of the relative intensities of these ions in the reference spectrum: 20% if relative ion response is $<100\%$ of primary ion, 40% if relative ion response is between 100% and 200% of primary ion, 60% if relative ion response is between 200% and 300% of primary ion, 80% if relative ion response is between 300% and 400% of primary ion (Example: For an ion abundance of 50% in the reference, the corresponding abundance in a sample spectrum can range between 30% and 70%; for an ion abundance of 150% in the reference, the corresponding abundance in a sample can range between 110% and 190%).

- 14.4.4 Structural isomers that produce very similar mass spectra must be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The Manual Integration SOP 1255 is used for guidance in the integration of these two peaks and for other situations that may require analyst efforts to correct computer generated integrations that are not technically sound.
- 14.4.5 Identification is hampered when sample components are not resolved chromatographically or co-elute and produce mass spectra containing ions contributed by more than one compound. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of compound spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds, but each compound spectrum will contain extraneous ions contributed by the coeluting compound.
- 14.4.6 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:
- 14.4.6.1 Molecular ions present in the reference spectrum must be present in the sample spectrum.
 - 14.4.6.2 Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.
 - 14.4.6.3 Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- 14.5 Data Interpretation - Quantitative Analysis
- 14.5.1 Once a compound has been identified, the quantitation of that compound must be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used must be the one nearest the retention time of that of a given compound. The responses of the internal standards in the samples must be within $\pm 40\%$ of the internal standard responses for the daily CCV sample.
- 14.5.2 The concentration in the sample is determined using the average response factor (RF) from initial calibration data (13.2). See Section 15.1.1 for the equations describing internal standard calibration.
- 14.5.3 Where applicable or required, the concentration of any non-target compounds (TICs) identified in the sample is estimated. The same formulae must be used with the following modifications: The areas A_x and A_{is} must be from the total ion chromatograms, and the RF for the compound must be assumed to be 1. The resulting concentration must be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.
- 14.6 Record the following information in the appropriate logbook or data file. Include any deviations from this procedure. Analyst initials, date of analysis, sample number or ID, initial sample volume or weight processed, calibration standard sample or solution identifier, QC sample or solution identifier, internal standard solution identifier, any dilution information, readings from support equipment, data file name, instrument method name, visual observations, and any other information as deemed necessary.

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- 14.7 Troubleshooting - The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that the analysis is performed, the daily calibration standard (CCV) must be evaluated to determine if the chromatographic system is operating properly. Questions that must be asked: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any major changes are made to the system (e.g. column changed or detector cleaned), recalibration of the system must take place.
- 14.8 Routine Maintenance - Record all maintenance, including the problem encountered, steps taken to correct the problem, and verification that the problem has been corrected (i.e. tune passed).
- 14.8.1 Bake out (using trap heating method) or change the Autocan Trap as needed (loss of sensitivity)
- 14.8.2 Clean the source on an as needed basis (loss of sensitivity or EMVoltage required to pass BFB and perform analyses is approaching 3000).
- 14.8.3 Repair leaks in the GC/MS system. The daily standard spectra auto-tune is very useful for detecting the need for source cleaning and/or leak repair.
- 14.8.4 Check the carrier gas supply; replace as necessary.
- 14.8.5 Replace purifier traps on carrier gas supply as needed- the use of indicating traps is a big help in determining the need for purifier replacement.
- 14.9 Sample Container Leak Test
The sample containers are pressurized to 30psia (15.3 psig) with zero air or nitrogen and allowed to stand on the cleanup assembly for a 12-hour period with the valves closed. After 12 hours, the container pressures are checked individually. If the canister pressure drops < 1.0 psi over the 12 hour period, the container is free of leaks. This test is performed when the container is cleaned and certified. If the client provides the sample container, a leak test will be performed when the container is cleaned and certified for return to the client.

15.0 DATA REDUCTIONS, CALCULATIONS, AND LOADING

- 15.1 The LIMS data system performs the calculations. Since each sample will have a preparation factor, this must be accounted for in the calculations.
- 15.1.1 The concentration in the sample is determined using the average response factor (RF) from the initial calibration data (13.2) and the following equation. The equation is based upon equivalent sample sizes of 500cc volume trapped.

$$\text{Concentration (ppb)} = \frac{A_x * C_{is} * PF}{A_{is} * RF}$$

where:

A_x = Area of the characteristic ion for the compound being measured

C_{is} = Concentration of the specific internal standard (10ppbv for this method)

A_{is} = Area of the characteristic ion for the specific internal standard

RF = average response factor for compound being measured

PF = Preparation factor as defined in 14.1.2.

If another sample volume is analyzed, adjust the concentration using the factor: 500cc / volume sampled cc.

15.2 If appropriate, recalculate the concentration of the compound in the sample using the equations in Secs. 15.2.1, 15.2.2, and 15.2.3.

15.2.1 The concentration of the compound in the sample is ppbv. If conversion to $\mu\text{g}/\text{m}^3$ is required, calculate as follows:

$$\text{Concentration } (\mu\text{g}/\text{m}^3) = \frac{\text{Conc. (ppbv)} * \text{MW}}{24.45}$$

Where:

24.45 is the volume in L of 1 mole of an ideal gas at 25 °C and standard pressure.

Conversion of L to m^3 is cancelled out by conversion of mg to μg .

MW is the molecular weight

15.3 The procedure for uploading data into the LIMS system is detailed in SOP 1400 LIMS.

16.0 METHOD PERFORMANCE

16.1 Demonstration of Capability (DOC)

All parameters of interest must meet the method acceptance criteria before actual sample analysis begins. See SOP 1230 Training for the procedure to perform and document the DOC. The DOCs for the analysts performing this method are located in the analysts' training form folders located in the QA office files.

The QC reference sample used is the LCS solution (see Section 10.7). The QC reference sample is made using stock standards prepared independently from those used for calibration. For this analysis, the LCS is the ICV standard which has a nominal concentration of 1-4 times the RL for each compound. This analysis is performed in quadruplicate using 4 separately prepared canisters. For each analyte calculate the mean recovery (X) and the standard deviation (s). Note: X must be within 30 % of the spike value, s must be less than 30 % of X and the mean recovery must be in the range of 70 to 130%. If X and s and %R for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual x or %R falls outside the range for accuracy or any individual s exceeds the precision limit, then the system performance is unacceptable for that analyte and corrective action must be taken.

Continuing Demonstration of Capability will be documented with four consecutive Laboratory Control Samples (LCS) or acceptable PT sample results. This must be documented annually.

16.2 Comparison to Reference Method Data

EPA Method TO-15 provides guidance for the establishment of control limits for the LCS QC samples. Recovery limits of 70% to 130% are provided as initial benchmarks for performance (EPA Method TO-15 Section 11.4.2). These limits are designed for a wide variety of organic test methods. Individual compound performance may vary.

16.3 In-House Control Limits

Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data is compared to the specifications of the reference method. If the in-house limits are within the specifications of the reference method, the control limits are updated in LIMS. If the in-house limits are not within specifications, an investigation is performed to determine the cause(s) of the problem and a corrective action is completed. The analysis may continue until enough data points are collected to regenerate new control limits. Any QC data generated outside of reference method limits during that time frame is flagged.

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained. After the analysis of at least 20 laboratory control samples, calculate the average percent recovery (R) and the standard deviation of the percent recovery (S).

Control limits for method parameters are generated by the QC staff generates the method parameters and distributed to the analysts via updates to the LIMS control charts. The control limits are calculated based on in-house performance data. At a minimum, these limits are reviewed annually.

17.0 POLLUTION PREVENTION

The preparation of excessive volumes of laboratory reagents and standards should be avoided so that waste and potential for pollution are minimized. Samples, reagents and standards must be disposed in compliance with the lab waste disposal program, SOP 1130 Waste Management.

Uncontaminated paper waste, glass and cans should be separated for recycling. Laboratory staff is required to protect the laboratory and our clients' business information when disposing of recycled paper or waste from the facility.

18.0 DATA ASSESSMENT AND CRITERIA FOR QUALITY CONTROL MEASURES

Data assessment includes review of: proper sample condition, preservation, and storage; analysis within holding time limitations; deviations from the SOP, evaluation of performance based on in-house control limits, reference method limits or project specific limits.

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation. The data review is conducted according to SOP 1250 Data Review.

18.1 Blanks

The method blank must contain less than the reporting limit for all target compounds. If the blank exceeds these limits, the source of contamination must be investigated and corrective actions taken. Re-analyze the method blank. If after re-analysis, the blank criteria is still exceeded, stop the analysis prior to sample analyses and correct the problem. Consult with the department manager for guidance. Refer to a client specific QAPP for additional guidance, if applicable.

If samples were analyzed with a contaminated method blank during unattended automated analysis, affected samples must be reanalyzed or data must be appropriately qualified if:

- 1) The concentration of a targeted compound in the blank is at or above the reporting limit as established by the SOP or by regulation, AND is greater than 1/10 of the amount measured in any sample.
- 2) The blank contamination otherwise affects the sample results as per the test method requirements or the individual project data quality objectives.

18.2 Laboratory Control Samples (LCS/LSCD)

The results of the individual batch LCS are calculated in percent recovery (%R) and compared to established acceptance criteria. Current acceptance criteria used are 70 - 130%; in-house LCS limits may be developed. For LCS Duplicate (LSCD), recovery limits must be met and the Relative Percent Difference (RPD) must be less than 25%. If the LCS is outside the acceptance criteria the analytical system is "out of control". Any affected samples associated with an out of control LCS must be reprocessed and reanalyzed or the results reported with appropriate data qualifiers. If after re-analysis the control criteria has not been met, the entire analysis batch must be re-analyzed. Always refer to a client specific QAPP for additional guidance.

18.3 Surrogates

Not applicable to this method.

18.4 Duplicates

The results from Laboratory Duplicates are designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). See the QAM, Section 5.4 for the calculation for RPD.

Replicate samples will be analyzed whenever the client provides them. However, when they are not, then the LCS/LCSD pair must be analyzed per batch of 20 incoming samples. In both cases, reproducibility performance is defined as $\pm 25\%$ RPD.

Note: It is an option to analyze the same can as a duplicate. The same acceptance criteria would apply.

For duplicates results outside established criteria corrective action must be documented or the data reported with appropriate data qualifying codes.

18.5 Matrix Spikes

The results from MS/MSDs are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). See the STAT Quality Assurance Manual, Section 5.4, for the calculation for RPD. Results are compared to established acceptance criteria, currently $\pm 30\%$ %R and $\pm 25\%$ % RPD. For matrix spikes outside established criteria, corrective action must be documented or the data reported with appropriate data qualifying codes.

18.6 Internal Standards

The results of the individual Internal Standard compounds on all samples, blanks, and spikes are calculated in percent recovery (%R) compared to the daily CCV sample and are compared to established acceptance criteria. If the internal standard recovery is outside the acceptance criteria, see Table 7, corrective action may be taken.

Table 7 Internal Standards

INTERNAL STD COMPOUND	CRITERIA (%)
Chlorobenzene-d ₅	60 to 140
Bromochloromethane	60 to 140
1,4-Difluorobenzene	60 to 140

18.7 Other

The laboratory will analyze standard reference materials and/or participate in relevant performance evaluation studies quarterly.

19.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

The process for handling unacceptable and out of control data is found in SOP 230 Corrective Action.

If the CCV, MB, LCS/LCSD, lab duplicate, MS/MSD or internal standard recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the samples is suspect and is only reported for regulatory compliance purposes with the appropriate corrective action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. Final data results must be qualified in the client report for reported results not meeting the laboratory-defined criteria.

- 1) Review standards preparation logbooks. Check all calculations and ensure dilution factors are properly recorded.
- 2) Re-prepare the suspected standard or QC sample to identify possible preparation errors of the standard or QC sample.
- 3) Re-Analyze the samples when the CCV or LCS is not within acceptable limits, or the internal standard fails.
- 4) Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

20.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Every effort is made to prevent problems from occurring. When out of control or unacceptable data occurs the first option is to identify the problem and reanalyze the samples within the holding times. When this is not possible, the QA Manager and/or the Laboratory Director reviews data and discusses options with the client. Reanalysis or reporting the data with qualification are alternatives. Out of control or unacceptable data reported to the client must include the data qualifier, flag and discussion on the rationale for reporting.

Holding time exceedence, improper preservation and improper sample condition or storage are noted on the corrective action form and included on the final report.

Review the CCV standard response, LCS result, and internal standard recovery for acceptable performance for each batch of samples. Record any trends or unusual performance on a corrective action form. Final data results must be qualified in the client report for results not meeting the laboratory-defined criteria.

Manual integration must be minimized for standards and the method blank. Routine manual integration of the same parameters indicates a system performance problem. Correct this problem or note in the instrument analysis logbook the suspected causes for routine manual integration. Sign and date all manually integrated chromatograms.

20.1 The process for handling unacceptable and out of control data is found in the Laboratory QAM Section 11. The reporting of data that is out of control must be approved and documented by Quality Assurance Manager and either the Technical Manager or the Laboratory Director.

20.2 Client Requested Modifications:

20.2.1 Clients must request modifications from the laboratory SOP in writing to the lab.

20.2.2 The lab director, technical manager and quality assurance manager will evaluate the requested client deviations, determine the feasibility of the deviation and the potential effects on the data.

20.2.3 If it is determined that the lab will perform the requested deviations, the lab director, technical manager and quality assurance manager will decide if a method validation study is required.

20.2.4 The designated project manager will retain all documentation concerning the requested deviation, including all correspondence with the client, in the client folder.

20.2.5 The final analytical report must include the statement "*This report has analyses performed using client requested modifications*".

21.0 WASTE MANAGEMENT

The STAT Analysis Corporation SOP 1130 Waste Disposal identifies proper waste management practices for the chemicals and biological materials used in this procedure. Samples are stored and discarded in accordance with SOP 1130 Waste Disposal.

22.0 REFERENCES

- 22.1 USEPA, 40CFR Part 136 Appendix B – Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11.
- 22.2 Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, 1999.
- 22.3 Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, 1999.
- 22.4 Quality Assurance Manual, STAT Analysis Corporation
- 22.5 SAP 003 Chemical Hygiene Plan
- 22.6 SOP 230 Corrective Action
- 22.7 SOP 1000 Control and Use of Laboratory Notebooks
- 22.8 SOP 1010 Analytical Standards and Reagents Receipt and Preparation
- 22.9 SOP 1040 General Laboratory Practices
- 22.10 SOP 1130 Waste Disposal
- 22.11 SOP 1210 Method Detection Limits, Limits of Detection, Limits of Quantitation
- 22.12 SOP 1230 Training
- 22.13 SOP 1250 Data Review
- 22.14 SOP 1255 Manual Integration
- 22.15 SOP 1330 Purchasing
- 22.16 SOP 1400 LIMS
- 22.17 Manufacturers' Equipment Instruction Manuals
- 22.18 NIST Library of Reference Spectra, 2002

23.0 FORMS, FIGURES, TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS OR VALIDATION DATA

Attachment 1: Changes from Revision 03

Attachment 2: MDLs And Reporting Limits for TO-14A and TO-15 Volatile Compounds

Attachment 3: Characteristic Ions For Volatile Compounds

Attachment 4: Volatile Internal Standards with Corresponding Compounds Assigned for Quantitation

Attachment 1: Changes from Revision 03

- Added analysis from glass jars.
- Clean canisters are evacuated to <0.05psia (2.6mm Hg), previous revision <0.7psia.
- Allows sample and standard preparation with nitrogen.
- Allows sample analysis without pressurizing incoming canister.
- GC/MS and Autocan operating conditions.
- Clean canister certification batch requires only CCV verification.
- Changes to requirements of characteristic ions.
- Section 12.6 revised to require MD/MSD when requested by the customer or periodically in support of the QC program.
- Revised Section 16 to clarify the procedure and acceptance criteria for DOC.
- Section 18.5 revised to provide guidance regarding MS/MSD data that does not meet established acceptance criteria.
- Editorial changes.

Attachment 2
MDLs and REPORTING LIMITS FOR TO-14A and TO-15 VOLATILE COMPOUNDS

COMPOUND NAME	TO-14	TO-15	MDL, ppbv	Lower Reporting Limit, ppbv	Upper Reporting Limit, ppbv
1,1,1-Trichloroethane	X	X	0.0038	0.2	50
1,1,2,2-Tetrachloroethane	X	X	0.0044	0.2	50
1,1,2-Trichloroethane	X	X	0.0065	0.2	50
1,1-Dichloroethane	X	X	0.0106	0.2	50
1,1-Dichloroethene	X	X	0.0087	0.2	50
1,2,4-Trichlorobenzene	X	X	0.0286	0.2	50
1,2,4-Trimethylbenzene	X	X	0.0110	0.2	50
1,2-Dibromoethane	X	X	0.0044	0.2	50
1,2-Dichlorobenzene	X	X	0.0114	0.2	50
1,2-Dichloroethane	X	X	0.0074	0.2	50
1,2-Dichloropropane	X	X	0.0185	0.2	50
1,3,5-Trimethylbenzene	X	X	0.0083	0.2	50
1,3-Butadiene		X	0.0410	0.2	50
1,3-Dichlorobenzene	X	X	0.0089	0.2	50
1,4-Dichlorobenzene	X	X	0.0158	0.2	50
1,4-Dioxane		X	0.0263	0.5	50
2-Butanone		X	0.1185	0.5	50
2-Hexanone		X	0.1065	1.0	50
4-Ethyltoluene		X	0.0069	0.2	50
4-Methyl-2-pentanone		X	0.0647	1.0	50
Acetone	X	X	0.4963	2.0	50
Benzene	X	X	0.0081	0.2	50
Benzyl chloride	X	X	0.0629	0.5	50
Bromodichloromethane		X	0.0037	0.2	50
Bromoform		X	0.0036	0.5	50
Bromomethane	X	X	0.0468	0.5	50
Carbon disulfide		X	0.0041	0.2	50
Carbon tetrachloride	X	X	0.0253	0.2	50
Chlorobenzene	X	X	0.0050	0.2	50
Chloroethane	X	X	0.0526	0.2	50
Chloroform	X	X	0.0041	0.2	50
Chloromethane	X	X	0.0274	0.5	50
cis-1,2-Dichloroethene	X	X	0.0156	0.2	50
cis-1,3-Dichloropropene	X	X	0.0269	0.2	50
Cyclohexane		X	0.0269	0.2	50
Dibromochloromethane		X	0.0200	0.2	50
Dichlorodifluoromethane	X	X	0.0061	0.2	50
Ethyl acetate		X	0.0647	0.5	50
Ethylbenzene	X	X	0.0052	0.2	50
Freon-113	X	X	0.0061	0.2	50
Freon-114	X	X	0.0113	1.0	50
Heptane		X	0.0298	0.2	50
Hexachlorobutadiene	X	X	0.0133	0.2	50

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Attachment 2 (continued)
MDLs and REPORTING LIMITS FOR TO-14A and TO-15 VOLATILE COMPOUNDS

COMPOUND NAME	TO-14	TO-15	MDL, ppbv	Lower Reporting Limit, ppbv	Upper Reporting Limit, ppbv
Hexane		X	0.0423	0.5	50
Isopropyl alcohol		X	0.0594	1.0	50
m,p-Xylene	X	X	0.0109	0.4	100
Methyl tert-butyl ether		X	0.0075	0.2	50
Methylene chloride	X	X	0.1232	2.0	50
Naphthalene		X	0.0389	0.2	50
o-Xylene	X	X	0.0189	0.2	50
Propene		X	0.0548	2.0	50
Styrene	X	X	0.0077	0.2	50
Tetrachloroethene	X	X	0.0047	0.2	50
Tetrahydrofuran		X	0.0186	0.5	50
Toluene	X	X	0.0049	0.2	50
trans-1,2-Dichloroethene		X	0.0109	0.2	50
trans-1,3-Dichloropropene	X	X	0.2000	0.2	50
Trichloroethene	X	X	0.0500	0.2	50
Trichlorofluoromethane	X	X	0.0049	0.2	50
Vinyl Acetate		X	0.1132	2.0	50
Vinyl chloride	X	X	0.0153	0.2	50
Xylenes, total	X	X	0.0425	0.6	150

Attachment 3: CHARACTERISTIC IONS FOR VOLATILE COMPOUNDS

#	Compounds	Retention Time (min.)	Primary Ion	Secondary Ion(s)
2	Propene	3.514	41	42, 39
3	Dichlorodifluoromethane	3.617	85	87
4	Chloromethane	3.818	50	52
5	Freon-114	3.965	85	135
6	Vinyl Chloride	4.081	62	64
7	1,3-Butadiene	4.245	54	39, 53
8	Bromomethane	4.532	94	96
9	Chloroethane	4.727	64	66
10	Ethanol	4.915	45	46
11	Acrolein	5.227	56	55
12	Acetone	5.354	43	58
13	Trichlorofluoromethane	5.513	101	103
14	Isopropyl Alcohol	5.629	45	43, 59
15	1,1-Dichloroethene	6.017	61	96, 63
16	Dichloromethane	6.232	49	84, 86
17	Carbon Disulfide	6.470	76	44
18	Freon-113	6.501	101	151, 85
19	trans-1,2-Dichloroethene	7.074	61	96, 98
20	1,1-Dichloroethane	7.257	63	65
21	MTBE	7.312	73	57
22	Vinyl Acetate	7.397	43	86
23	2-Butanone	7.592	43	72
24	cis-1,2-Dichloroethene	7.976	61	96, 98
26	Hexane	8.183	57	86, 41
27	Ethyl Acetate	8.165	61	45, 70
28	Chloroform	8.232	83	85
29	Tetrahydrofuran	8.549	42	72, 71
30	1,2-Dichloroethane	8.854	62	64, 49
31	1,1,1-Trichloroethane	9.080	97	99, 61
32	Benzene	9.470	78	51, 77
33	Carbon Tetrachloride	9.598	117	119, 121
34	Cyclohexane	9.714	84	41, 69
35	1,2-Dichloropropane	10.165	63	41, 76
36	Bromodichloromethane	10.323	83	85
37	Trichloroethene	10.372	130	95, 60
38	1,4-Dioxane	10.342	88	58, 43
39	Methyl Methacrylate	10.518	69	99, 59
40	Heptane	10.646	43	100, 57
41	cis-1,3-Dichloropropene	11.110	75	110
42	4-Methyl-2-Pentanone	11.134	43	58, 100
43	trans-1,3-Dichloropropene	11.567	75	77
44	1,1,2-Trichloroethane	11.732	97	83, 61
45	Toluene	12.000	91	92
46	2-Hexanone	12.213	43	100, 58
47	Dibromochloromethane	12.372	129	127, 131
48	1,2-Dibromoethane	12.609	107	109
49	Tetrachloroethene	13.054	166	129, 94

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#	Compounds	Retention Time (min.)	Primary Ion	Secondary Ion(s)
51	Chlorobenzene	13.701	112	77, 114
52	Ethylbenzene	14.079	106	91
53	m&p-Xylene	14.268	106	91
54	o-Xylene	14.743	106	91
55	Xylene, total	14.743	106	91
56	Bromoform	14.323	173	91
57	Styrene	14.621	104	78
58	1,1,2,2-Tetrachloroethane	14.713	83	85
59	4-Ethyltoluene	16.115	120	105
60	1,3,5-Trimethylbenzene	16.206	120	105
61	1,2,4-Trimethylbenzene	16.706	120	105
62	Benzyl Chloride	16.864	91	126
63	1,3-Dichlorobenzene	16.889	146	111, 75
64	1,4-Dichlorobenzene	16.974	146	111, 75
65	1,2-Dichlorobenzene	17.413	146	111, 75
66	1,2,4-Trichlorobenzene	19.895	180	182, 145
67	Naphthalene	20.059	128	129, 127
68	Hexachlorobutadiene	20.651	225	260, 190
	Internal Standards:			
1	Bromochloromethane	8.110	49	128, 130
25	1,4-Difluorobenzene	9.762	114	63
50	Chlorobenzene-d5	13.658	117	119, 82

Attachment 4: Volatile Internal Standards with Corresponding Compounds Assigned for Quantitation

Bromochloromethane (I)	1,4-Difluorobenzene (I)	Chlorobenzene-d5 (I)
Propene	Hexane	Chlorobenzene
Dichlorodifluoromethane	Ethyl acetate	Ethylbenzene
Chloromethane	Chloroform	m,p-Xylene
Freon-114	Tetrahydrofuran	o-Xylene
Vinyl chloride	1,2-Dichloroethane	Bromoform
1,3-Butadiene	1,1,1-Trichloroethane	Styrene
Bromomethane	Benzene	1,1,2,2-Tetrachloroethane
Chloroethane	Carbon tetrachloride	4-Ethyltoluene
Ethanol	Cyclohexane	1,3,5-Trimethylbenzene
Acrolein	1,2-Dichloropropene	1,2,4-Trimethylbenzene
Acetone	Bromodichloromethane	Benzyl chloride
Freon-11	Trichloroethene	1,3-Dichlorobenzene
Isopropyl alcohol	1,4-Dioxane	1,4-Dichlorobenzene
1,1-dichloroethene	Methyl Methacrylate	1,2-Dichlorobenzene
Dichloromethane	Heptane	1,2,4-Trichlorobenzene
Carbon disulfide	cis-1,3-Dichloropropene	Naphthalene
Freon-113	4-Methyl-2-pentanone	Hexachlorobutadiene
trans-1,2-Dichloroethene	trans-1,3- Dichloropropene	
1,1-Dichloroethane	1,1,2-Trichloroethane	
Mtbe	Toluene	
Vinyl Acetate	2-Hexanone	
2-Butanone	Dibromochloromethane	
cis-1,2-Dichloroethene	1,2-Dibromoethane	
	Tetrachloroethene	

(I) = Internal Standard